NASA TECHNICAL NOTE

NASA TN D-2577

NASA TN D-2577

PERFORMANCE OF SEVERAL ABLATION MATERIALS EXPOSED TO LOW CONVECTIVE HEATING RATES IN AN ARC-JET STREAM

by Marvin B. Dow and William D. Brewer

Langley Research Center
Langley Station, Hampton, Va.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • JANUARY 1965

PERFORMANCE OF SEVERAL ABLATION MATERIALS EXPOSED TO LOW CONVECTIVE HEATING RATES IN AN ARC-JET STREAM

By Marvin B. Dow and William D. Brewer

Langley Research Center
Langley Station, Hampton, Va.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Office of Technical Services, Department of Commerce,
Washington, D.C. 20230 -- Price $2.00

PERFORMANCE OF SEVERAL ABLATION MATERIALS EXPOSED TO LOW CONVECTIVE HEATING RATES IN AN ARC-JET STREAM

By Marvin B. Dow and William D. Brewer
Langley Research Center

SUMMARY

An investigation was made of the performance of eleven different types of ablation materials bonded to beryllium or aluminum backup plates. Thirty-four specimens fabricated as flat square sections with a nominal side dimension of 5 inches (12.70 cm) were exposed to convective heating rates of less than 10 Btu/(sq ft)(sec) (0.114 MW/m^2) for exposure times in excess of 200 seconds in the 2500-kilowatt arc jet at the Langley Research Center. Some of the specimens were tested in air and others, in a mixture of air and nitrogen. The back-surface temperature response, the mass-distribution loss of ablation materials, and the depth of thermal degradation are presented. The performance of each material during testing is also discussed.

A comparison of measured and calculated back-surface temperature responses is presented for some of the ablation materials tested in the arc jet. Calculations were made for those materials for which data concerning physical and thermal properties were available. Reasonable agreement was obtained between experimental and calculated temperature responses.

Photographs of ablation materials which experienced reentry on the afterbody of the spacecraft of the Project Mercury mission MA-8 are shown. A comparison is made of the physical appearance of identical materials tested in flight and in the arc jet. This comparison indicates that, despite the more severe oxidation in the arc-jet stream, certain materials developed the same type of physical defects in both the reentry and arc-jet environments.

INTRODUCTION

The performance of several thermal-protection materials during reentry was investigated in a flight test conducted by the Manned Spacecraft Center. For this flight test, several ablation materials were bonded to the beryllium shingles on the afterbody of the spacecraft of the Project Mercury mission MA-8. Although the heat-transfer rates encountered by the materials were not severe, the test did provide an actual flight environment for comparing the materials.

As a means of acquiring information on the relative performance of the materials to aid in interpretation of flight-test results, specimens of the same

materials were exposed to convective heating in an electric-powered arc-jet facility at the Langley Research Center. Other materials, not flight tested, were also tested in the ground facility.

Results obtained from the arc-jet tests are presented herein in terms of material thermal response, mass-distribution loss, thermal degradation, and visual observation of material behavior. A comparison is made of the physical appearance of the materials tested in the arc jet and in flight. Also presented are comparisons of measured and calculated back-surface temperatures for four of the test materials.

The units for the physical quantities used in this paper are given both in the U.S. Customary Units and in the International System of Units, SI (ref. 1). An appendix is included to explain the relationship between these systems of units.

ARC-JET SPECIMENS AND TEST APPARATUS

The test specimens consisted of an ablation material bonded to a metal backup plate. Completed test specimens were furnished by seven different suppliers. The specimen fabrication techniques for each ablation material were developed by its supplier. It was specified that the mass distribution of the thermal-protection system, including the ablation material, the bond material, and the moisture-proof protective coating, if required, be approximately 0.5 lb/sq ft (2.44 kg/m^2).

General information concerning the arc-jet specimens is summarized in table I, which includes the trade name of the ablation materials, the name of the firm which supplied each ablation material, and the number of specimens furnished for arc-jet testing. Hereafter, the materials are identified by the letters appearing with the trade names in table I.

Arc-Jet Specimens

The arc-jet specimens were flat and square, with a nominal side dimension of 5 inches (12.70 cm). The ablation materials were bonded to beryllium backup plates, 0.10 inch (0.25 cm) thick, except those made with materials F and G. (See table I.) These specimens were fabricated with 6061-T6 aluminum-alloy backup plates which were 0.156 inch (0.396 cm) thick. All arc-jet specimens, except the four made from material H, were fabricated with only one test material per specimen. The ablation material applied to one-half of each specimen of material H contained a fiber-glass mesh reinforcement; this material was cast and adhesively bonded to the beryllium backup plate. The ablation material applied to the other half of the plate was spray applied and adhered from self-bonding. The test-material thickness on both halves of the specimen was the same. The bond line between the two materials was oriented parallel to the direction of the stream flow.

Arc-jet specimens of each type of ablation material are shown prior to testing in the photographs of figure 1. A photograph of a specimen of material J prior to testing is unavailable, but its appearance is identical to the specimens of material I.

A detailed summary of the mass distributions of the ablation materials is shown in table II for each arc-jet specimen. The values shown are based on the nominal density and the thickness data furnished by the supplier.

Five chromel-alumel thermocouples were attached to the uncoated side of the metal backup plate for measuring its temperature response. The location of these thermocouples is shown in figure 2. The three thermocouples near one edge of the specimen were used to monitor the temperature of the metal backup plate in the region of the most severe heating. The temperatures indicated by the two thermocouples located near the center of the backup plate were considered the prime data. The thermocouple arrangement shown in figure 2 was used for all arc-jet specimens, except the specimens of material H. For these specimens one prime-data thermocouple was placed on the center line parallel to the interface of each of the two test materials. Thermocouple outputs were recorded on an oscillograph.

Test Apparatus

Arc-jet facility.- The test specimens, which were 5 inches (12.70 cm) square, were exposed to the high-temperature gas stream produced by the 2500-kilowatt arc jet at the Langley Research Center. This facility produces a subsonic gas stream at atmospheric pressure with a static temperature of about 6,800° F (4033° K) and an enthalpy of approximately 3,000 Btu/lb (6.96 MJ/kg) at the exit of the nozzle. Construction details and operation of the facility are described in reference 2.

Specimen mounting and test fixture.- Hat-shaped steel clips were welded to the metal backup plates in the position shown in figure 2 to provide a means of attaching the specimens to the test fixture. The instrumented specimens were fastened by means of screws to sheet-steel holders. An assembly of the specimen in the holder is shown in figure 3. The specimens were mounted in the holders so that the bond line between the ablation material and the metal backup plate was at the elevation of the flanges of the holder. Only the ablation material protruded out of the holder. The space between the edges of the metal backup plate and the holder was completely filled with silicone rubber. The back face of the metal backup plate was insulated with fibrous insulation, and the steel attachment clips were insulated from the holder by a sheet of asbestos. Therefore, the only metal-to-metal contact was that resulting from the four screws which connected the attachment clips and the holder.

A water-cooled test fixture was used to position the test specimens in the arc-jet stream and is shown, in the photograph of figure 4, with a specimen installed. The test fixture was constructed of copper and consisted of a cylindrical leading edge with a radius of 2 inches (5.08 cm) and a 9.5° half-angle wedge-shaped afterbody. The outer surface and edges of the ablation materials

were exposed to the arc-jet stream in the same manner as were the flight specimens bonded to the afterbody shingles of the MA-8 spacecraft.

TEST CONDITIONS AND PROCEDURE

Test Conditions

An estimate of the convective heating rates to be encountered by the flight specimens on the afterbody of the MA-8 spacecraft during reentry was obtained from temperatures measured on the uncoated beryllium afterbody shingles of the MA-5 spacecraft during reentry. The calculated "cold-wall" heating rates corresponding to the temperatures measured on the MA-5 spacecraft are shown plotted against reentry time in figure 5. Inasmuch as limitations of the arc-jet facility precluded exact duplication of the MA-5 heat pulse, two constant levels of heating rate were used in the arc-jet tests, as shown in figure 5. The time of exposure at each heating rate was adjusted to give the same total heat input as the flight-reentry heat pulse. The arc-jet simulation of reentry heating shown in figure 5 will hereinafter be referred to as the simulated pulse.

The arc-jet heating rates shown in figure 5 represent repeated measurements of the heating rate at the center of a metal calorimeter, 5 inches (12.70 cm) square, which was substituted for the test specimen. In order to obtain heat-transfer rates of the level required for simulation, it was necessary to use an arc-jet nozzle 12 inches (30.48 cm) in diameter and to position the center of the test specimens 22 inches (55.88 cm) above the nozzle exit. The combination of the large-diameter nozzle and the distance between the nozzle exit and the test specimen caused the arc-jet stream to be somewhat unsteady in the test region. This unsteadiness in turn caused some variation in the measured values of the heating rate. The arc-jet heating-rate curves in figure 5 are drawn as bands in order to reflect the degree of uncertainty in the measured values. The change in level of the heating rate shown in figure 5 was obtained by changing the mass flow of gas through the nozzle.

The available equipment did not permit a determination of the enthalpy of the test stream 22 inches (55.88 cm) above the nozzle exit. The measured value of the test-stream enthalpy near the nozzle exit was 3,000 Btu/lb (6.96 MJ/kg). Diffusion into the stream by unheated surrounding air and some loss of energy in the gas stream due to radiation produced a marked decrease in the stream enthalpy at the position of the test specimen. The enthalpy of the stream at the position of the test specimen was estimated to be 1,000 Btu/lb (2.32 MJ/kg).

Test Procedures

In general, two types of tests were performed on each ablation material. In one type of test, the material was exposed only to the simulated heat pulse shown in figure 5. In the other type of test, the high-level portion of the heat pulse was continued until a temperature of 600° F (589° K) was measured by the prime-data thermocouples. A photograph of a typical specimen during testing

is shown in figure 6. Also shown are pertinent details of the arc-jet facility and the test apparatus.

Nitrogen was used in place of air in the arc-jet stream for tests of several specimens to determine the effects of oxidation on some of the ablation materials. Previous tests (ref. 3) have indicated that oxidation can have a considerable effect on the performance of certain ablation materials in the arc-jet facility used in the present investigation.

Samples of gas were obtained from the nitrogen arc-jet stream 22 inches (55.88 cm) above the nozzle exit. These samples were analyzed to determine the amount of air which had mixed with the nitrogen stream. The analysis indicated that the test stream contained approximately 12 to 16 percent oxygen which had diffused into the stream from the surrounding air.

At the completion of the testing, the specimens were weighed to determine the weight loss of the ablation material, and the specimens were sectioned to determine the depth of thermal degradation in the test material at the center of the specimen. The maximum temperature of the metal backup plate and the time required to reach this temperature, measured from the start of the test, were determined from the temperature records of the prime-data thermocouples for those specimens exposed to the simulated pulse.

RESULTS AND DISCUSSION

The results of all arc-jet tests are summarized in table III. Included in table III are the specimen exposure times, the temperature of the metal backup plate at specific times during the test, the unit weight loss of the ablation material, and the depth of thermal degradation in the ablation material.

The low- and high-level heating rates shown in table III refer to the heating rates of the simulated pulse shown in figure 5. Exposure to high-level heating in excess of 55 seconds indicates that the test was continued until the metal backup plate reached 600° F (589° K). In most cases the simulated pulse consisted of 160 seconds of low-level heating followed by 55 seconds of high-level heating; the exception to this in the case of a few specimens was due to changes in the arc-jet heating rate which required an adjustment in exposure time. The effect on backup plate temperatures of such variation in the simulated pulse was small. The temperature of the metal backup plate at the end of the simulated pulse is either the temperature at the termination of heating or the temperature at a time corresponding to the end of the simulated pulse for those specimens for which the high-level heating was continued until the metal backup plate reached 600° F (589° K). It should be noted that for some ablation materials the temperature of the metal backup plate reached 600° F or more from the simulated pulse alone. The maximum temperature and the time to reach maximum temperature give an indication of the amount of heat stored in the test material and the time required for this heat to penetrate the self-insulation provided by the ablation material.

Care must be exercised in estimating the performance, based on test results in ground facilities, of ablation materials in flight applications. Ablation materials tested in air at enthalpies considerably lower than the predicted flight enthalpy are subjected to disproportionately severe oxidation. An unpublished analysis indicates that simulation of char performance in low-enthalpy arc jets requires testing at greatly reduced oxygen concentrations. For the present tests, a reduction in the oxygen concentration of the test stream to a level acceptable for adequate simulation was not possible. This impossibility was because the specimen was located at a point in the arc-jet test stream where considerable mixing with ambient air occurred. Even when 100-percent nitrogen was supplied to the arc jet, the oxygen concentration at the test position of the specimens was from about 12 to 16 percent. This concentration was substantially higher than the 3 percent or less, by weight, required for good simulation of char performance.

The ablation materials on the arc-jet specimens were not subjected to the noise and vibration of launching, the near-vacuum conditions of orbital flight, or the high-velocity stream flow. However, for the reason stated in the preceding paragraph, oxidation during the arc-jet tests of these materials was much more severe than would be anticipated during reentry on the afterbody of the MA-8 spacecraft. This severity of oxidation would penalize the performance of those materials which are particularly susceptible to oxidation.

Temperature Histories Obtained From Arc-Jet Tests

The temperature histories of the specimens tested in the arc-jet facility are shown in figure 7. The temperatures shown on these curves are an average of the measurements from the two prime-data thermocouples located near the center of each specimen. In most cases, these temperatures show good agreement between specimens of the same ablation material. This agreement indicates uniform material properties and satisfactory duplication of test conditions. The temperature histories of the specimens tested in a mixture of nitrogen and air indicate to some extent the effect of oxidation on these particular materials. The obvious exception to good agreement between temperature histories obtained during different tests of similar materials can be seen in figure 7(g). In this instance, there is a considerable difference in the measured temperatures on two specimens tested in the same gas stream for the same length of time. This difference in temperatures is discussed in the following section.

Ablation-Material Performance During Testing and Physical Appearance After Testing

Visual observations during testing and motion-picture records indicated that the various ablation materials exhibited marked differences in performance during exposure to arc-jet heating. Also, the physical appearance of the various ablation materials at the completion of testing differed widely. The photographs of figure 8 show a specimen of each ablation material after exposure to the simulated pulse.

Phenolic nylon.- Combustion, producing visible flames, occurred on the surface of the phenolic nylon during exposure to the low-level heating. The char layer which formed developed a pattern of large-grained cracks resembling "mud flat" cracks over the entire surface. The edge of the ablation material which was closest to the arc-jet nozzle experienced the most severe heating and was beveled by oxidation. Oxidation of the material occurred at an increased rate during high-level heating, with the width of the cracks in the char gradually becoming wider because of localized combustion in the cracks. No blistering or separation of material occurred during testing. The phenolic-nylon material was also tested in an arc-jet stream of reduced oxygen concentration. The same crack pattern formed in the char but oxidation was less severe, as evidenced by narrow cracks and absence of edge beveling.

Figure 8(a) shows the phenolic-nylon material after a simulated pulse in air. The crack pattern, which is probably due to shrinkage of the char, is evident in the photograph. The char layer adhered to the unpyrolized material, with no visible distortion or separation; however, the char was friable and could be rubbed off with moderate pressure. This material experienced a 46- and 33-percent weight loss from exposure to the simulated pulse in air and in a stream of reduced oxygen concentration, respectively.

Material A.- This material changed in color from a light brown to a dark brown during exposure to the low-level heating. There was no visible evidence of combustion during this period. The high-level heating caused visible combustion on the surface with a gradual removal of material which exposed what appeared to be a matting of quartz fibers. Some cracks and local checking appeared on the surface during the high-level heating.

Figure 8(b) shows the material after exposure to the simulated pulse. The surface of the specimen after testing was flat, with no distortions or serious defects other than the cracks and checking noted previously. The outer surface of the material was hard, and considerable pressure was required to rub charred material from the surface. The weight of material lost from the two specimens exposed to the simulated pulse was in close agreement and was approximately 30 percent. The temperature of the beryllium backup plate at the end of the simulated pulse showed some variation among the four test specimens. This variation in temperature may have been caused by the distribution of the quartz in the ablation material.

Material B.- Approximately 10 seconds after the beginning of exposure to low-level heating, the entire surface of material B began to oxidize, and visible flames were produced. The oxidation of this material appeared to occur at a faster rate than for any other material tested. Exposure to the high-level heating increased the rate of oxidation, and during this period particles of material were blown from the surface by the test stream.

Material B is shown after testing in figure 8(c). This ablation material was almost totally consumed during simulated pulse heating as evidenced by a material weight loss of about 90 percent. The light-colored material on the specimen surface which can be seen in figure 8(c) is probably bond material, inasmuch as material B was completely consumed in this area. The material on the remainder of the specimen resembled soot in appearance and strength.

Material C.- Combustion, producing visible flames, occurred on the surface of the specimens of material C during low-level heating. Blisters and fissures appeared in the material near the end of the low-level heating period. When the heating was increased to the high level, the rate of combustion increased appreciably. The fissures in the material widened, and localized burning occurred in the region of the fissures. The ablation material separated from the beryllium backup plate in the region of the most severe heating, which was the edge of the specimen nearest the arc-jet nozzle.

Figure 8(d) shows the specimen after exposure to the simulated pulse. Material C was seriously affected by oxidation with nearly a 50-percent material weight loss. The blisters and fissures which appeared during testing are visible in figure 8(d). The beryllium backup plate reached a temperature of 600° F (589° K) before the end of the simulated pulse. (See table III.)

Material D.- During exposure to the low-level heating in air, material D gradually changed in color from red to black. There was no visible evidence of surface oxidation. When the heating was increased to the high level, the surface of the material oxidized and visible flames were produced. The specimen surface gradually changed in color from black to white. There was no blistering, separation, or loss of material from the honeycomb during the test period. Material D was also exposed to the simulated pulse in an arc-jet stream of reduced oxygen content. The appearance and performance of the material during this test were identical to the test in air.

Figure 8(e) shows material D after having been tested in air. The white surface layer which formed was extremely fragile and easily separated from the material beneath and would most likely have been removed by a higher velocity test stream. This material showed good oxidation resistance, as evidenced by the weight loss of about 11 percent. The amount of material weight loss during the test at reduced oxidation concentration was not determined because of loss of the degraded layer before weighing could be accomplished; however, the depth of thermal degradation in the two tests was the same.

Material E.- During exposure to low-level heating, material E changed in color from a medium brown to black, with no visible evidence of combustion. The high-level heating caused visible combustion on the surface. There was no blistering, separation, or loss of material from the honeycomb during testing. This material was also tested in an arc-jet stream of nitrogen and air and evidenced less visible combustion during high-level heating in this test stream, but otherwise it performed in the same manner as in the air tests.

Material E, after exposure to the simulated pulse in air, is shown in figure 8(f). The surface of the material was rough but regular, with the honeycomb cell walls apparently having oxidized at a slower rate than the ablation material. The weight of material lost during the simulated pulse in air was about 45 percent of the weight before testing and about 30 percent for the corresponding test in nitrogen and air.

Material F.- Low-level heating in air caused combustion of material F, with visible flames over the entire surface of the material. Cracks formed in the surface of the material, and localized combustion occurred in the regions of the

cracks. High-level heating caused increased oxidation on the surface of the material. Cracks penetrating the entire depth of the ablation material developed, and the material blistered and separated from the aluminum backup plate in the region of the most severe heating.

Tests of the material in an arc-jet stream of reduced oxygen concentration resulted in less severe oxidation, even though flames were visible during both low- and high-level heating. The cracking and separation of material was also less severe than for the tests in air.

The ablation material F is shown in figures 8(g) and 8(h) after exposure to the simulated pulse in air and at reduced oxygen concentration, respectively. The cracks and separations of the material, which are similar in appearance to those which occurred with material C, can be seen in the photographs. The char layer produced by the tests in air extended in depth to the aluminum plate, with no unpyrolized material remaining. The material experienced a 66-percent weight loss during tests in air.

The difference in the temperature histories between specimens 1 and 2 of material F noted in the section entitled "Temperature Histories Obtained From Arc-Jet Tests" is difficult to explain. (See fig. 7(g).) The two specimens were both tested in an arc-jet stream at reduced oxygen concentration for identical test periods. Since the gas supplied to the arc jet was 100-percent nitrogen in both tests, it appears unlikely that there was any significant difference in the oxygen content of the test stream. Also, there was no indication of thermocouple malfunction, since all five thermocouples on each specimen were in working order and gave comparable temperatures. An error in thermocouple-data reduction does not explain the differences in the depth of thermal degradation and in the ablation-material weight loss between the two specimens. Specimen 1 of material F, which had the lowest back-surface temperatures, experienced a weight loss of 36 percent, whereas specimen 2, which had back-surface temperatures that agreed very closely with those of the specimens tested in air, experienced a weight loss of 58 percent.

Material G.- Specimens of material G differed from all others tested in that the rectangular fiber-glass honeycomb cells had low-density insulation between the metal backup plate and the ablation material. The ablation material was applied in a thin layer at the top of the cells and across the exterior surface of the specimens to give a smooth surface. When this material was exposed to low-level heating during a simulated pulse in air, a thin char layer formed at the surface, oxidized, and produced visible flames. The burning stopped after a few seconds, and the material in each cell appeared to expand outward with continued exposure. With the application of high-level heating, the thin char layer was rapidly oxidized and the insulation material in the honeycomb cells shrank away from the cell walls. Near the end of the high-level heating period, a portion of the insulation material in some honeycomb cells blew out and beads of glassy material were seen to form on the outer edges of most honeycomb walls. The formation of beads indicated that high local heating on the exposed edges was melting the glass in the honeycomb. When exposed to a simulated pulse in the arc-jet stream of reduced oxygen concentration, material G behaved as it did in air during the low-level heating. However,

exposure to the high-level heating produced no noticeable change in the appearance of the material. The char layer was not oxidized, nor was there any melting of the edges of honeycomb walls.

Figures 8(i) and 8(j) show material G after exposure to a simulated pulse in air and at reduced oxygen concentration, respectively. The glass beads which formed on the edges of the honeycomb walls are visible in figure 8(i). During tests in air, the ablation material G was completely consumed, and the specimens experienced a 56-percent weight loss. Material G tested at reduced oxygen concentration experienced a weight loss of about 45 percent.

Material H.- The protective coating on material H burned off quickly on exposure to low-level heating. With the protective coating gone, the interface in the center of the specimen between the differently applied sections of material H was evident. The low-level heating produced no visible reactions or changes in the material. The temperature of the beryllium backup plate rose continuously during low-level heating, as shown in figure 7(i). When the heating was increased to the high level, the surface of the specimen began to burn with green-colored flames. The half of the specimen covered by the sprayed-on and self-bonding material H developed an overall small-grain crack pattern; several large blisters which buckled the surface of the material also appeared. The section of adhesively bonded material H covering the other half of the specimen developed a large-grain crack pattern over most of the surface. Near the end of the high-level heating period, separation of this material from the beryllium backup plate occurred in the region of maximum heating.

The photograph of figure 8(k) shows a specimen of material H after exposure to the simulated pulse in air. The crack patterns and blistering are evident in the photograph. The surface cracking probably resulted from shrinkage of the thermally degraded material, and the blistering was probably caused by gas, formed during heating, which was unable to escape to the surface. Similar blisters developed in material H in other tests in the 2500-kilowatt arc jet at the Langley Research Center. (For example, see fig. 6(h) of ref. 4.) During exposure to the simulated pulse, the material experienced a 38-percent loss in weight.

The temperatures of the beryllium backup plate at the center of the two differently applied sections of material H were in close agreement. The difference in temperature between the two sections was less than 10° F (6° K), except at the conclusion of the tests of specimens 3 and 4, which were tested for longer times than the simulated pulse. The temperature difference at the conclusion of the testing of these two specimens was about 30° F (17° K), with the highest temperature occurring under the adhesively bonded material H. It is believed that separation of the material at the most severely heated edge was responsible for this divergence in temperatures.

Material I.- The surface of material I burned with visible flames upon initial exposure to low-level heating, but after a few seconds the visible flames disappeared. The material in the honeycomb cells appeared to swell outward, and in several individual cells the material was pushed completely out of the cell. Near the end of the low-level heating period the thermally degraded surface layer appeared to separate from the material beneath and to produce large

blistered regions extending across several cells. When the heating was increased to the high level there was surface combustion, and visible flames were produced. Some of this material was blown completely out of several of the cells, and there was localized burning around such cell holes. Also, the material continued to separate into blistered regions in the same manner as during low-level heating.

Figure 8(l) shows a specimen of material I after a simulated pulse in air. Individual cells from which the material blew out are visible in the photograph. The thin white layer of thermally degraded material which developed during the tests was distorted and irregular; this layer was extremely fragile, however, and had testing been conducted with a higher velocity air stream, it is likely that this layer would have been removed. Beneath the thin residue, the pyrolyzed material was hard and tough, but irregular. Material I experienced a weight loss of about 10 percent during exposure to the simulated pulse, thus indicating good resistance to oxidation. Examination of the temperature curves shown in figure 7 shows that the back-surface temperature for this material was higher than for any other material tested at corresponding times up to about 140 seconds of exposure; however, between 140 seconds and 160 seconds the back-surface temperature appeared to be reaching an equilibrium value. When the heating rate was increased, the back-surface temperature again increased but at a slower rate than during the low-level heating. It is believed that the back-surface temperatures of this material were markedly influenced by the considerable expansion of this material during heating. Examination of a sectioned specimen indicated that the thickness of the material after testing was nearly twice that before testing. This increasing thickness resulted from the expansion of the material up and out of the honeycomb cells. The expansion of material I increased the material thickness and decreased its density, thus giving it better insulation properties. Therefore, the thermal performance appeared to improve with longer exposure.

Material J.- Certain modifications were made in the formulations of material I in an effort to prevent the material from blowing out of the honeycomb. Subsequent tests of two specimens of material J (modification of material I) revealed no such defects. Figure 8(m) shows a specimen of material J after exposure to a simulated pulse in air, and the absence of holes and irregularities is evident. As with material I, a fragile surface layer of thermally degraded material was produced which had little bonding to the hard rough pyrolyzed material beneath.

Comparison of Calculated and Experimental Temperature Responses for Arc-Jet Specimens

The equations of the numerical analysis of reference 5 have been programed for solution by a high-speed digital computer. This program was used to calculate the temperature response of the backup plate for some of the arc-jet specimens during exposure to the simulated pulse. Calculations were made for specimens of materials B, C, H, and I. These materials were selected as being representative of the various materials tested. The temperature histories from figures 7(c), 7(d), 7(i), and 7(j) for specimens exposed to the simulated pulse have been replotted in figure 9 for comparison with calculated temperature

responses. The calculated and experimental temperatures show reasonable agreement, particularly in view of the uncertainties in the inputs for the calculations.

Examination of figure 9(d) shows that reasonable agreement was obtained for calculated and experimental temperatures during the low-level heating of material I. During the high-level heating, the calculated temperatures are somewhat higher than the measured temperatures. This difference probably results from the previously discussed expansion of material I during heating. At the present time, there is no provision in the computer program for an increase in material thickness during heating.

For calculation purposes, it was assumed that the materials were exposed to a constant-level enthalpy of 1,000 Btu/lb (2.32 MJ/kg). This reduction in enthalpy from the 3,000 Btu/lb (6.96 MJ/kg) measured near the nozzle exit appeared reasonable because there was a 50- to 70-percent dilution with unheated air. In addition, there was some loss of energy in the gas stream because of radiation. The heating rates and exposure times used in calculations were those shown in figure 5 for the arc jet.

The major uncertainties in the calculations, insofar as material properties were concerned, were the specific heat of the gaseous products of pyrolysis and the thermal conductivity of the thermally degraded material or char. The majority of the data available on the materials for which calculations were made was obtained from measurements taken at room temperature. Thus, little information was available concerning the material properties at high temperatures. The values used in initial calculations were obtained by assuming that the properties varied in much the same manner as those of similar materials for which the properties are known functions of temperature.

Another source of disagreement between experimental and calculated temperatures may have resulted from variations in the heating rate at different positions on the test specimens. The computer program is capable of handling only one-dimensional heating. The material on the test specimens was not uniformly heated, inasmuch as the heating rates decreased with increasing distance from the arc-jet nozzle. Also, the metals (beryllium and aluminum) used as backup plates in fabricating the specimens have high values of thermal conductivity; therefore, the temperature at the center of the plates is influenced by nonuniform heating. Furthermore, it was assumed for calculation purposes that the metal backup plates were perfectly insulated, whereas, in fact, they did not have perfect insulation.

Comparison of Arc-Jet Specimens and Flight Specimens After Reentry

Specimens of each type of ablation material after the flight test on the MA-8 spacecraft are shown in figure 10. The photographs show the materials as they appeared on the beryllium afterbody shingles after spacecraft recovery. A comparison of the ablation materials which were flight tested and the arc-jet specimens in figure 8 indicates that the materials were less severely damaged in flight than in the arc-jet environment. This difference in damage may have resulted from unduly severe oxidation in the arc-jet environment. It is also

possible that the flight specimens did not receive heating as great as the arc-jet panels because of their position on the afterbody of the MA-8 spacecraft.

A comparison of the physical appearance of the specimens tested in flight (fig. 10) and the specimens tested in the arc jet (fig. 8) shows considerable similarity in the appearance of most of the materials. The similarity in appearance is apparent for phenolic nylon and for materials A, C, E, and H. There is some similarity between the appearance of the flight-tested specimen of material I (fig. 10(g)) and the specimen tested in the arc jet (fig. 8(ℓ)). The surface of the flight-tested specimens of material I was apparently discolored by deposition of its ablation products on the specimen surface or some other material which may have been ablation products from the forebody heat shield. In the region of most severe heating on the flight specimen, however, there are several honeycomb cells from which the material is gone. This type of failure was also observed during arc-jet tests. The specimen of material D tested in flight (fig. 10(e)) did not have the white friable surface layer that formed during the arc-jet tests of this material. This difference in appearance may have been caused by the degraded surface layer having been blown off the flight specimen, or the reentry heating may not have been sufficient to cause thermal degradation of the material. The appearance of the flight-tested material B (fig. 10(c)) is considerably different from the appearance of this material after arc-jet testing (fig. 8(c)). Much less of the material was consumed during the flight test, and the black sooty surface layer which developed during arc-jet testing is not present.

CONCLUDING REMARKS

An investigation was made of the performance of eleven different types of ablation materials bonded to beryllium or aluminum backup plates when tested in the 2500-kilowatt arc jet at the Langley Research Center, and the results are compared with identical materials tested in flight. These materials differed substantially in performance. Some of the materials withstood exposure to the arc-jet environment and developed few or no serious defects; other materials developed one or a combination of several severe defects, such as cracks, blisters, separations, and ejection of material from honeycomb cells. These defects would, if they occurred in flight, make the materials unfit for use as thermal protection on reentry vehicles. It is concluded that the arc-jet environment was unduly severe in regard to oxidation and that this adverse environment penalized the performance of some of the test materials.

Reasonable agreement was obtained between the measured and calculated back-surface temperature responses for those materials for which calculations were made. The use of the computer program for investigating the performance of heat-shielding materials depends upon the availability of detailed physical and thermal properties. The full utilization of the capability of the computer program used in the present investigation and other similar programs requires that much greater efforts be made by the developers of ablation materials to obtain reliable physical and thermal property data. Such data at high temperatures are especially needed.

The appearance of the ablation materials which were flight tested on the afterbody of the spacecraft MA-8 of the Project Mercury mission indicates that the materials were less severely damaged in the flight environment than in the arc-jet environment. It was, however, apparent that the materials which developed severe defects in the arc-jet environment also developed the same type of defects during reentry. The use of thermal protection materials for heat shielding during reentry will require that the overall performance of selected materials be investigated. Reliable protection will not be assured merely because the material demonstrates an ability to maintain a prescribed back-surface temperature, either in ground tests or by calculation.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., September 15, 1964.

APPENDIX

CONVERSION FACTORS

The International System of Units (SI) was adopted by the Eleventh General Conference on Weights and Measures, Paris, October 1960, in Resolution No. 12 (ref. 1). Conversion factors required for units used in this paper are presented in the following table:

Physical quantity	U.S. Customary unit	Conversion factor (a)	SI unit
Length	in.	0.0254	meters (m)
Mass distribution	lb/sq ft	4.88	kilograms/(meter)^2 (kg/m^2)
Heating rate	Btu/(sq ft)(sec)	1.135×10^4	watts/(meter)^2 (W/m^2)
Enthalpy	Btu/(lb)	2.32×10^3	joules/kilogram (J/kg)
Temperature	$^\circ$F + 460	5/9	$^\circ$Kelvin ($^\circ$K)

[a]Multiply value given in U.S. customary unit by conversion factor to obtain equivalent value in SI unit.

Prefixes to indicate multiples of units are:

milli (m)	10^{-3}
centi (c)	10^{-2}
kilo (k)	10^3
mega (M)	10^6

REFERENCES

1. Anon.: International System of Units - Resolution No. 12. NASA TT F-200, 1964.

2. Chapman, Andrew J.: An Experimental Evaluation of Three Types of Thermal Protection Materials at Moderate Heating Rates and High Total Heat Loads. NASA TN D-1814, 1963.

3. Dow, Marvin B.; and Swann, Robert T.: Determination of Effects of Oxidation on Performance of Charring Ablators. NASA TR R-196, 1964.

4. Dow, Marvin B.; Pittman, Claud M.; and Croswell, William F.: Thermal Performance and Radio-Frequency Transmissivity of Several Ablation Materials. NASA TN D-1896, 1964.

5. Swann, Robert T.; and Pittman, Claud M.: Numerical Analysis of the Transient Response of Advanced Thermal Protection Systems for Atmospheric Entry. NASA TN D-1370, 1962.

TABLE I.- SUMMARY OF SPECIMENS TESTED IN ARC JET

Source of specimen	Ablation material			Number of specimens
	Name (a)	Major constituents	Specific gravity	
Langley Research Center	Phenolic nylon[b]	47 percent powdered nylon 25 percent phenolic resin 23 percent phenolic microballoons 5 percent silica spheres	0.545	4
Chance Vought Corporation	Thermolite[b] (A)	Phenolic melamine plus quartz fibers	0.977	4
Avco Corporation	Avcoat II[b] (B)	Epoxy plus additives	0.962	2
	Avcoat 5026-22[b] (C)	Epoxy and phenolic microballoons plus additives	0.962	2
General Electric Company	ESM 1001[b] (D)	Modified silicone rubber plus additives in honeycomb[c]	0.882	2
	Series 500-27[b] (E)	Epoxy plus phenolic microballoons plus additives in honeycomb[c]	0.513	2
Martin Company	ESA 943 (F)	Epoxy plus silicone additives	0.946	4
	ESA 943 composite (G)	Honeycomb[d]: cells are partially filled (next to aluminum plate) with insulation of specific gravity from 0.289 to 0.310; remainder of each cell is filled with material F	0.513	4
Emerson Electric Manufacturing Company	Thermo-Lag T-500[b] (H)	Inorganic subliming salts plus phenolic binder	1.280	4
McDonnell Aircraft Corporation	S-3[b] (I)	White silicone and silica spheres plus additives in honeycomb[c]	0.890	4
	Modified S-3 (J)	Modification of material I	0.890	2

[a]Letters in parentheses are used to identify materials elsewhere in paper.
[b]Flight tested also.
[c]Hexagonal-cell fiber-glass honeycomb.
[d]Rectangular-cell fiber-glass honeycomb.

TABLE II.- SUMMARY OF THICKNESSES AND MASS DISTRIBUTIONS FOR SPECIMENS TESTED IN ARC JET

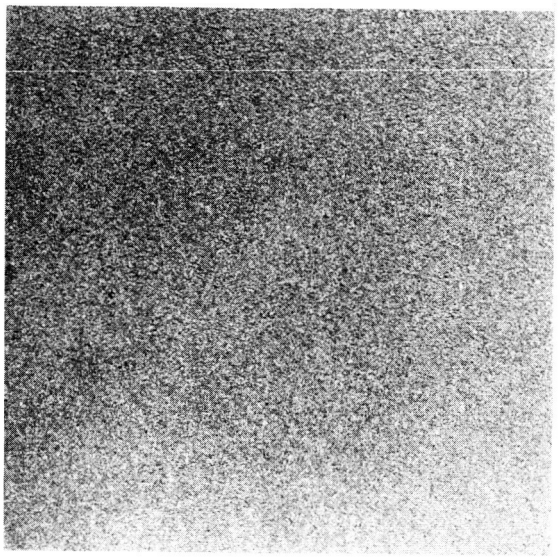
Ablation material							Bonding-material mass distribution		Protective-coating weight	Total specimen weight (a)	
Identification	Specimen number	Specific gravity	Thickness		Mass distribution						
			in.	mm	lb/sq ft	kg/m^2	lb/sq ft	kg/m^2		lb/sq ft	kg/m^2
Phenolic nylon	1	0.551	0.16	4.06	0.46	2.25	0.05	0.24	Negligible	1.51	7.37
	2	.534	.16	4.06	.45	2.20	.04	.20	↓	1.49	7.27
	3	.550	.16	4.06	.47	2.29	.06	.29		1.53	7.47
	4	.541	.17	4.32	.48	2.34	.06	.29		1.54	7.52
A	1	0.977	0.09	2.29	0.46	2.25	0.10	0.49	0	1.56	7.61
	2	↓	↓	↓	.44	2.15	.10	.49	↓	1.54	7.52
	3				.46	2.25	.12	.59		1.58	7.71
	4				.51	2.49	.13	.63		1.64	8.00
B	1	0.962	0.10	2.54	0.50	2.44	0.07	0.34	0	1.57	7.66
	2	.962	.10	2.54	.50	2.44	.07	.34	0	1.57	7.66
C	1	0.962	0.10	2.54	0.50	2.44	0.07	0.34	0	1.57	7.66
	2	.962	.10	2.54	.50	2.44	.07	.34	0	1.57	7.66
D	1	0.874	0.09	2.29	0.41	2.00	0.03	0.15	0	1.44	7.03
	2	.905	.09	2.29	.43	2.10	.03	.15	0	1.46	7.13
E	1	0.515	0.15	3.81	0.40	1.95	0.11	0.54	Negligible	1.51	7.37
	2	.510	.15	3.81	.40	1.95	.11	.54	Negligible	1.51	7.37
F	1	0.946	0.12	3.05	[b]0.50	2.44	(c)	(c)	0	[d]2.62	12.79
	2	↓	↓	↓	↓	↓	↓	↓	↓	↓	↓
	3										
	4										
G	1	[e]0.513	0.11	2.79	0.42	2.05	0.08	0.39	0	2.62	12.79
	2	↓	↓	↓	↓	↓	↓	↓	↓	↓	↓
	3										
	4										
H	1	1.28	0.09	2.29	0.50	2.44	0.08	0.39	Unknown	1.58	7.71
	2	↓	↓	↓	↓	↓	↓	↓	↓	↓	↓
	3										
	4										
I	1	0.890	0.11	2.79	0.51	2.49	0.07	0.34	0	1.58	7.71
	2	↓	↓	↓	↓	↓	↓	↓	↓	↓	↓
	3										
	4										
J	1	0.890	0.11	2.79	0.51	2.49	0.07	0.34	0	1.58	7.71
	2	.890	.11	2.79	.51	2.49	.07	.34	0	1.58	7.71

[a]Includes weight of metal backup plate but does not include unknown protective-coating weight.
[b]Includes mass distribution of bonding material.
[c]Weight included in mass distribution of ablation material.
[d]Metal backup plate is 6061-T6 aluminum, 0.156 inch thick.
[e]Combination of bonding material, honeycomb, and insulation.

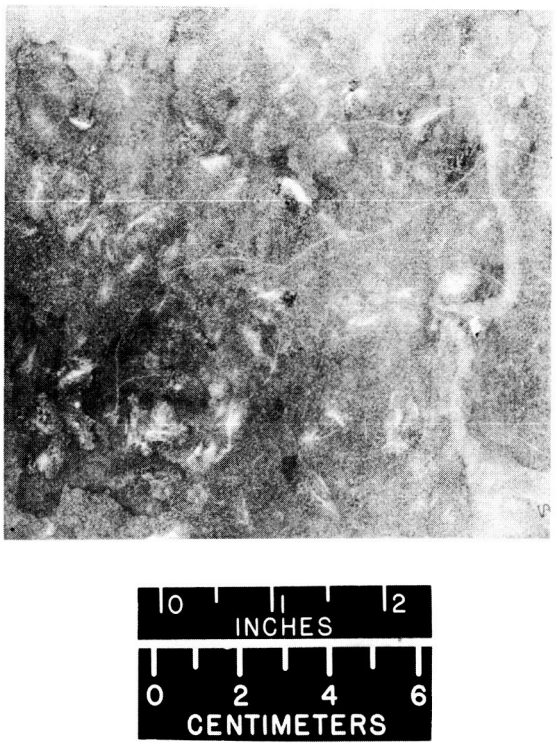
TABLE III.- ARC-JET TEST RESULTS

Material	Specimen number	Arc-jet exposure, sec		Thermal response of metal backup plate						Loss of ablation material		Depth of thermal degradation at center of specimen	
				From simulated pulse		Maximum temperature after pulse		Time to maximum temperature, sec	Time to 600° F (539° K), sec				
		Low-level heating	High-level heating	°F	°K	°F	°K			lb/sq ft	kg/m²	in.	mm
Phenolic nylon	1	160	48	459	511	516	542	241		0.28	1.37		
	2	↓	61	468	516					.30	1.46	0.08	2.03
	3		120	464	513				265	.33	1.61	.09	2.29
	[a]4		55	424	491	464	513	245		.20	.98	.06	1.52
A	1	160	55	512	540	553	563	233		0.18	0.88	0.05	1.27
	2	↓	55	565	569	608	593	233		.16	.78	.05	1.27
	3		85	536	553				232	.25	1.22	.07	1.78
	4		94	490	528				244	.26	1.27	.09	2.29
B	1	[b]145	70	588	582	615	597	231		0.46	2.25	0.10	2.54
	2	160	80	565	569				299	.49	2.39	.10	2.54
C	1	160	55	640	611	686	637	239	201	0.27	1.32	0.09	2.29
	2	160	55	649	616	688	638	242	200	.27	1.32	.08	2.03
D	1	160	55	579	577	618	599	237		0.04	0.20	0.02	0.51
	2	160	55	576	576	602	590	234				.02	.51
E	1	160	55	426	492	468	516	235		0.25	1.22	0.11	2.79
	2	160	55	411	484	458	510	249		.17	.83	.10	2.54
F	[a]1	160	55	408	482	493	529	229		0.18	0.88	0.05	1.27
	[a]2	↓	↓	576	576	587	582	220		.29	1.42	.07	1.78
	3			600	589	654	619	245	215	.34	1.66	.11	2.79
	4			580	578	641	612	246		.32	1.56	.11	2.79
G	[a]1	160	55	605	592	630	605	232	213	0.22	1.07	(c)	
	[a]2	↓	↓	560	567	586	581	237		.23	1.12	(c)	
	3			713	652	750	672	231	188	.28	1.37	(d)	
	4			690	639	746	670	233	195	.28	1.37	(d)	
H	1	160	48	529	549	551	561	222		0.21	1.03	0.06	1.52
	2	↓	55	509	538	530	550	231		.22	1.07	.06	1.52
	3		115	517	543				256	.36	1.76	.07	1.78
	4		115	505	536				257	.35	1.71	.06	1.57
I	1	160	55	482	523	538	554	240		0.06	0.29	0.04	1.02
	2	↓	130	484	524				263	.08	.39	.04	1.02
	3		48	465	514	522	546	240		.05	.24	.04	1.02
	4		120	482	523				260	.06	.29	.05	1.27
J	1	160	55	517	543	576	576	250					
	2	160	90	528	549				242				

[a]Tested in stream of nitrogen and air with oxygen concentration from about 12 to 16 percent, by weight.
[b]Low-level heating terminated too quickly because of experimental error.
[c]Thermal degradation indeterminate; material charred to insulation interface. (Original depth of material was not known.)
[d]Ablation material completely consumed with only insulation remaining.

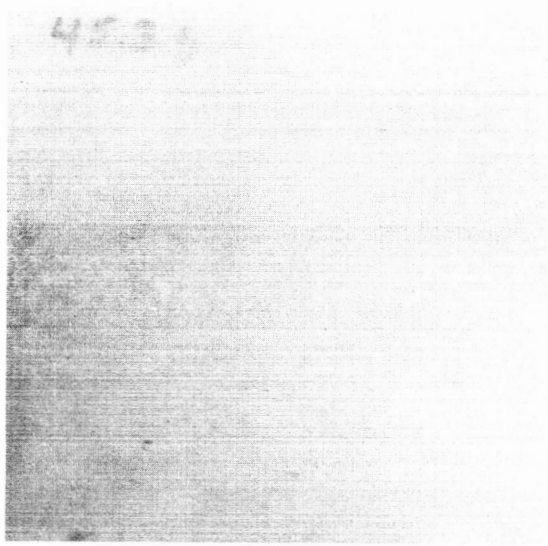


(a) Phenolic nylon.

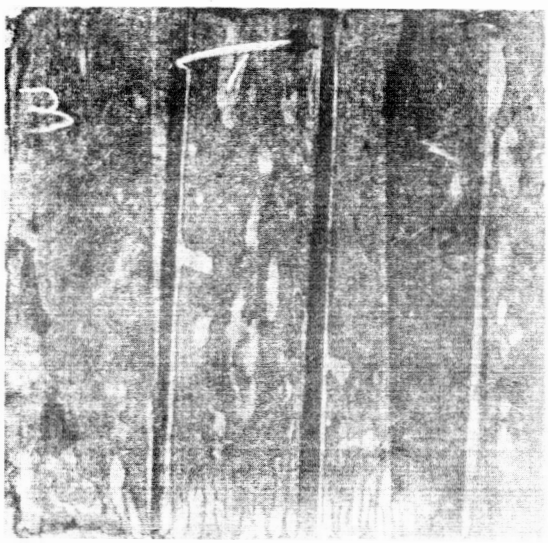


(b) Material A. L-64-8354

Figure 1.- Specimens before testing in arc jet.

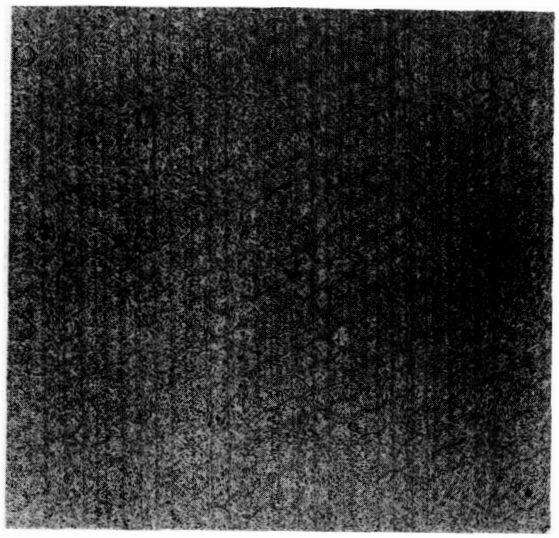


(c) Material B.

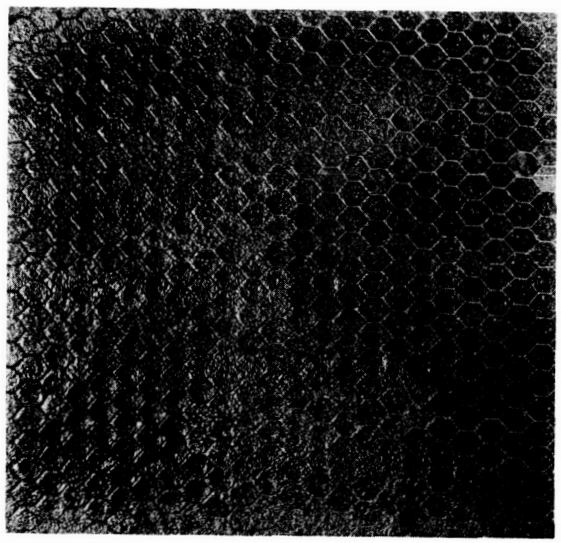


(d) Material C. L-64-8355

Figure 1.- Continued.

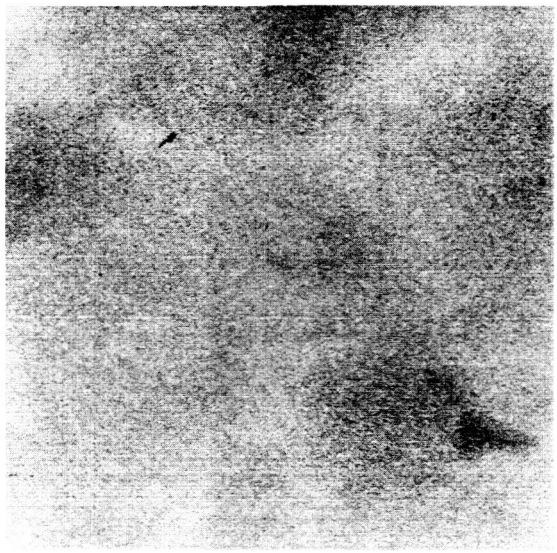


(e) Material D.

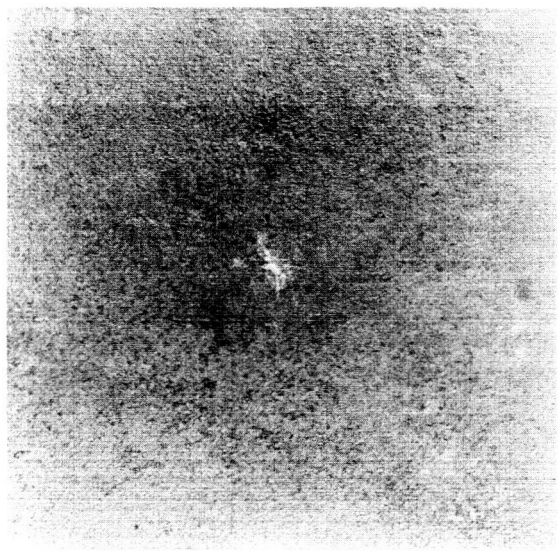


(f) Material E. L-64-8356

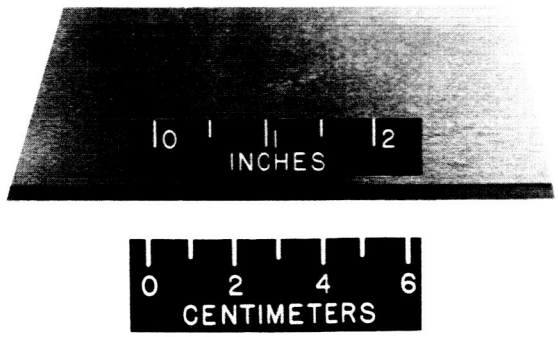
Figure 1.- Continued.



(g) Material F.



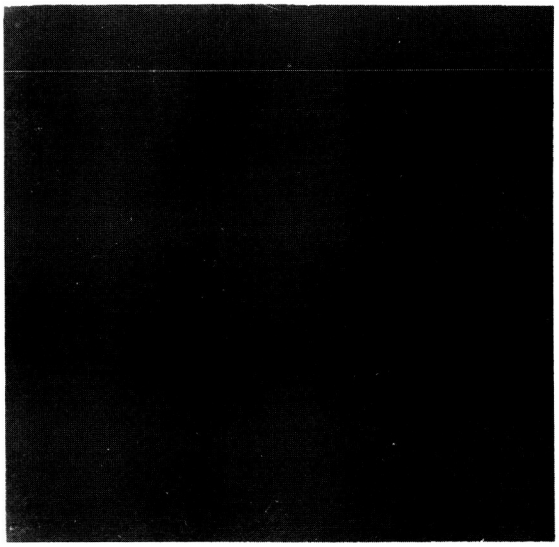
(h) Material G.



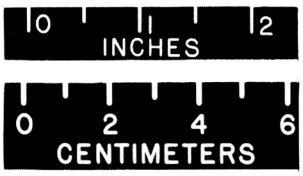
(i) Side view of material G.

L-64-8357

Figure 1.- Continued.



(j) Material H.



(k) Material I. L-64-8358

Figure 1.- Concluded.

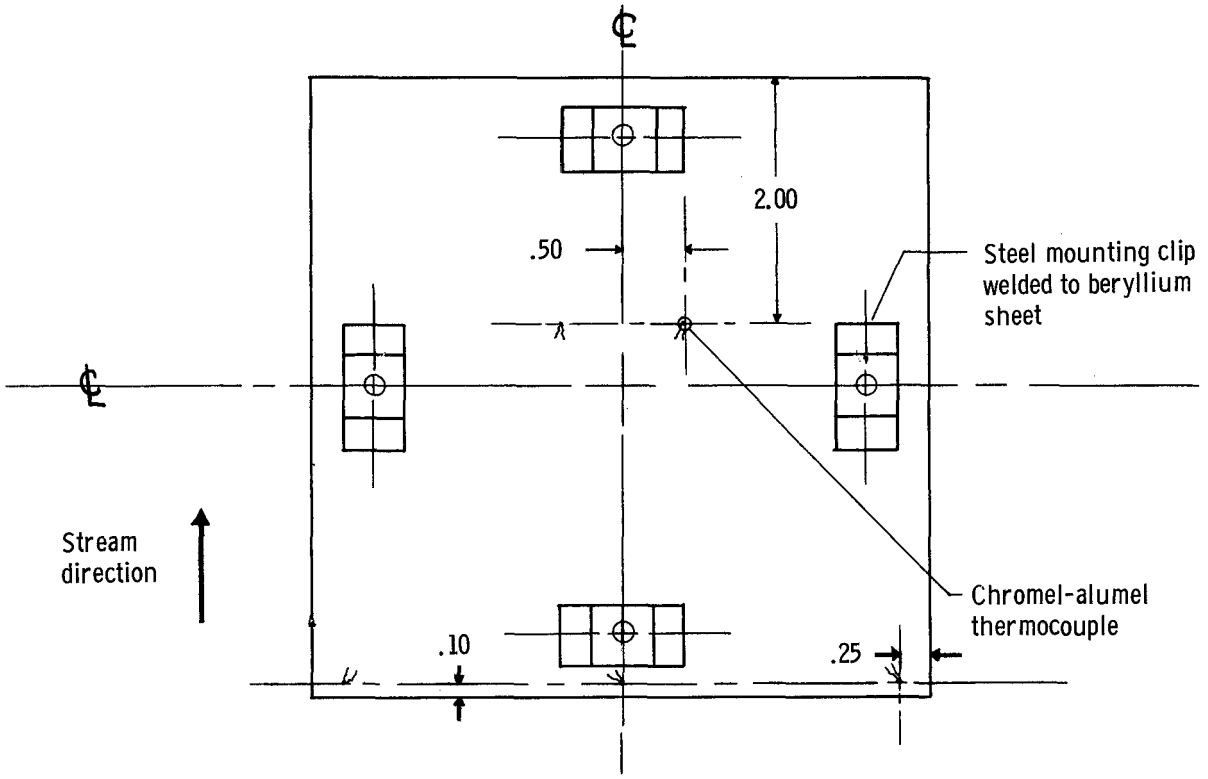


Figure 2.- Location of thermocouples and steel attachment clips on arc-jet test specimens. (All dimensions are in inches.)

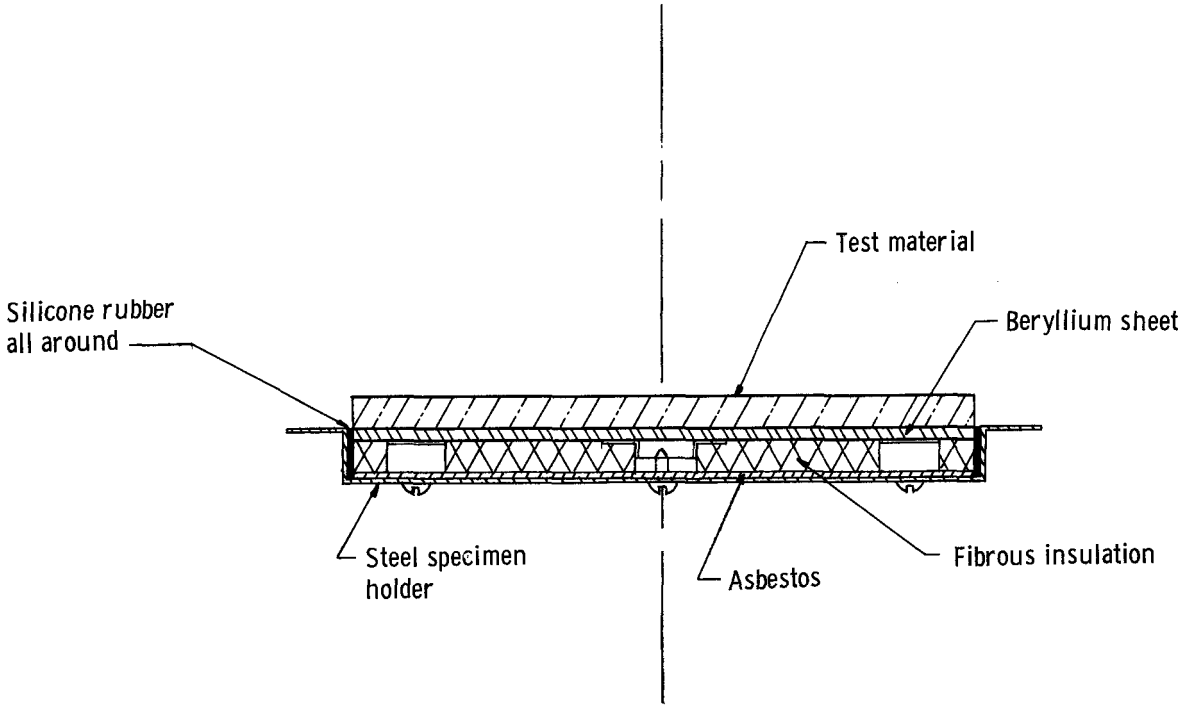
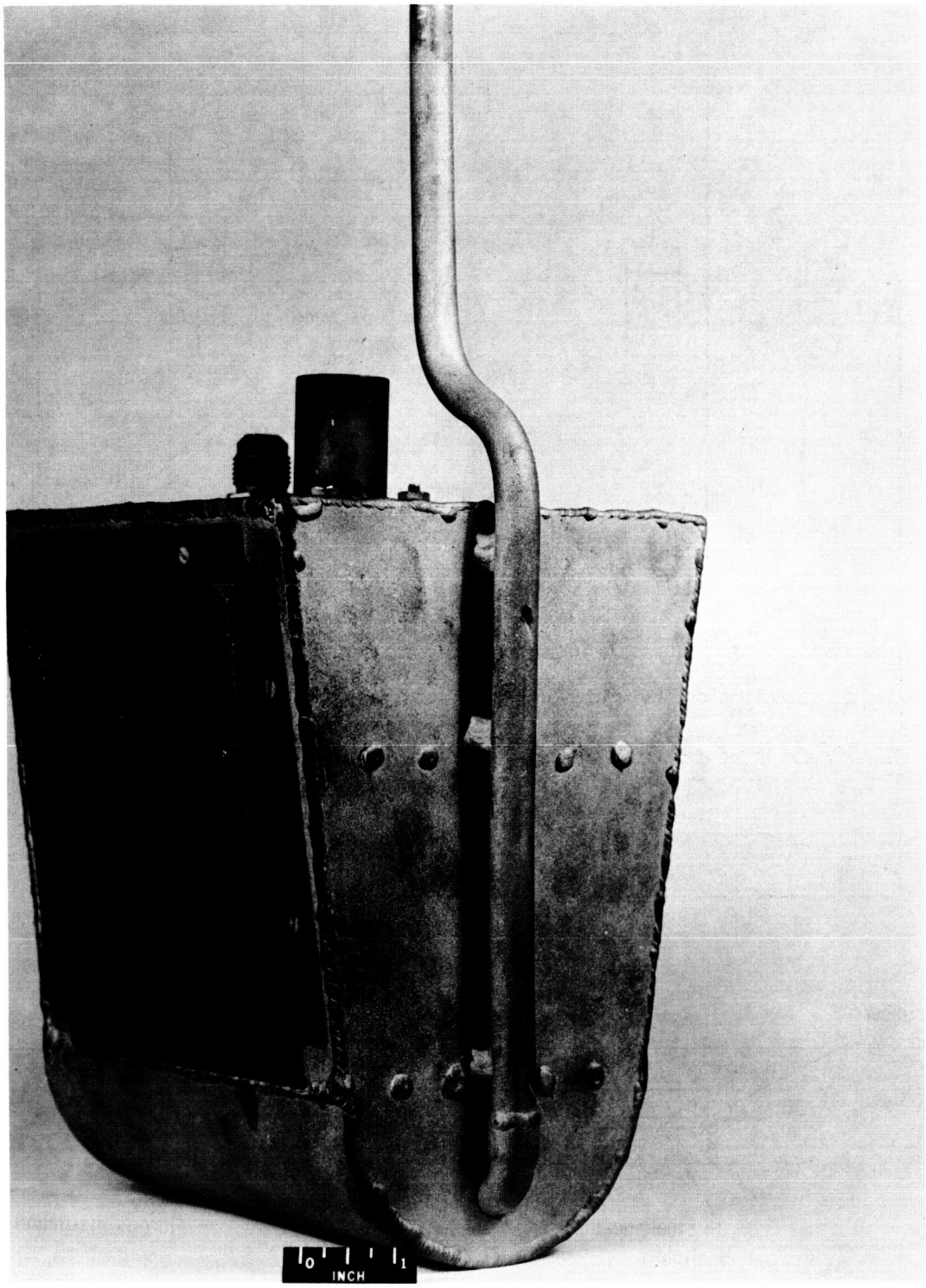


Figure 3.- Assembly of arc-jet test specimen in steel holder.



L-62-6385

Figure 4.- Water-cooled test fixture with test specimen attached.

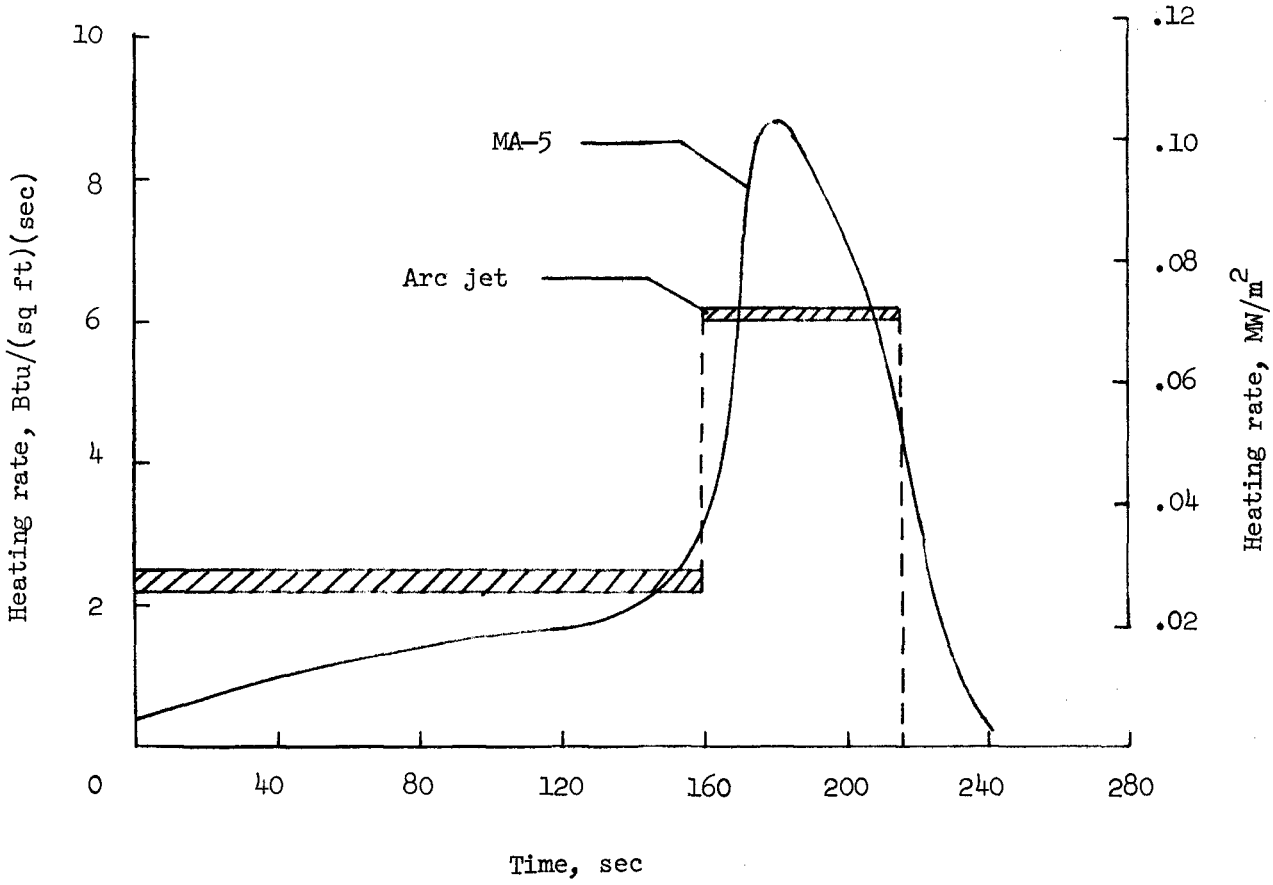


Figure 5.- Arc-jet simulation and MA-5 heating rates.

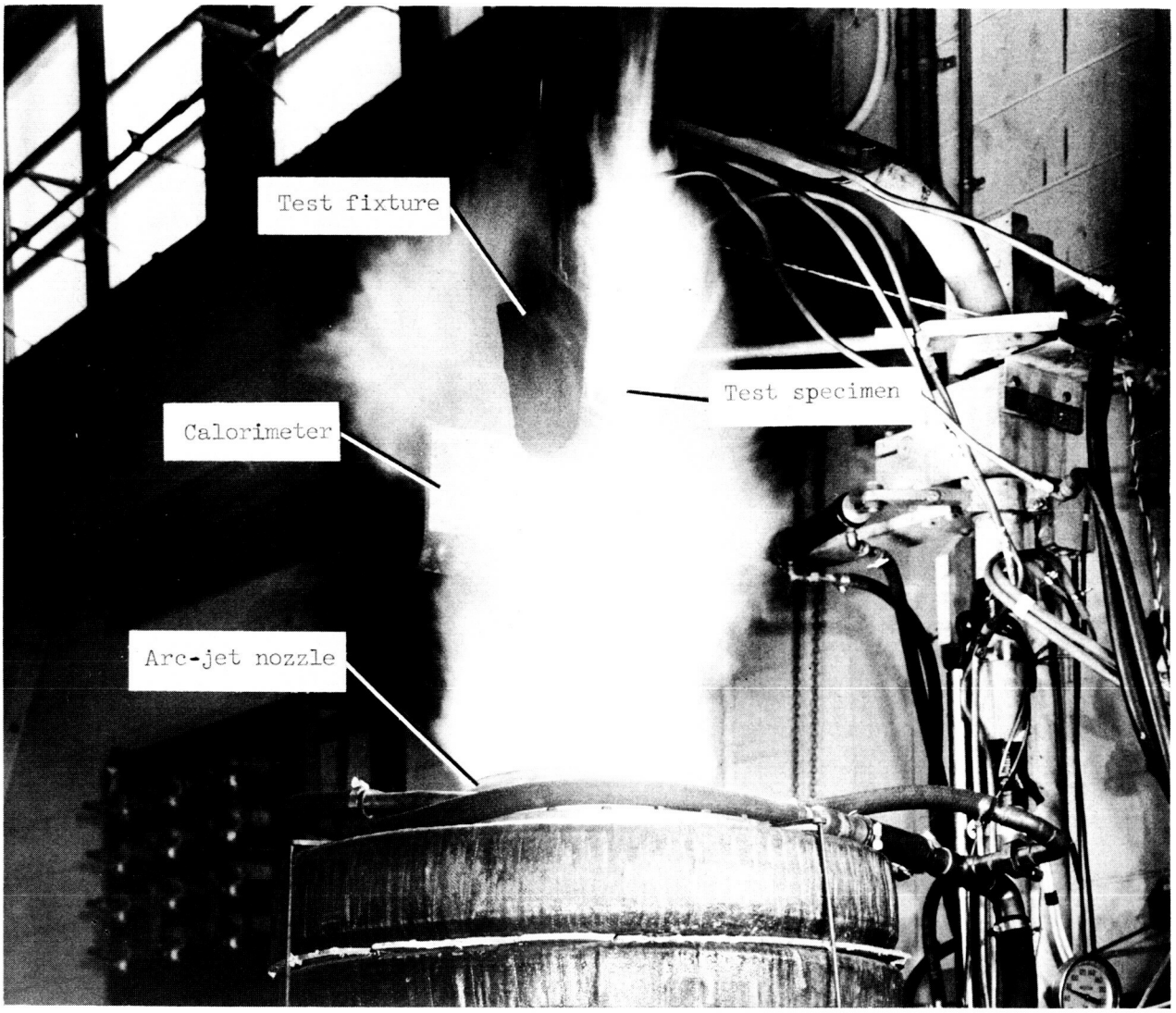
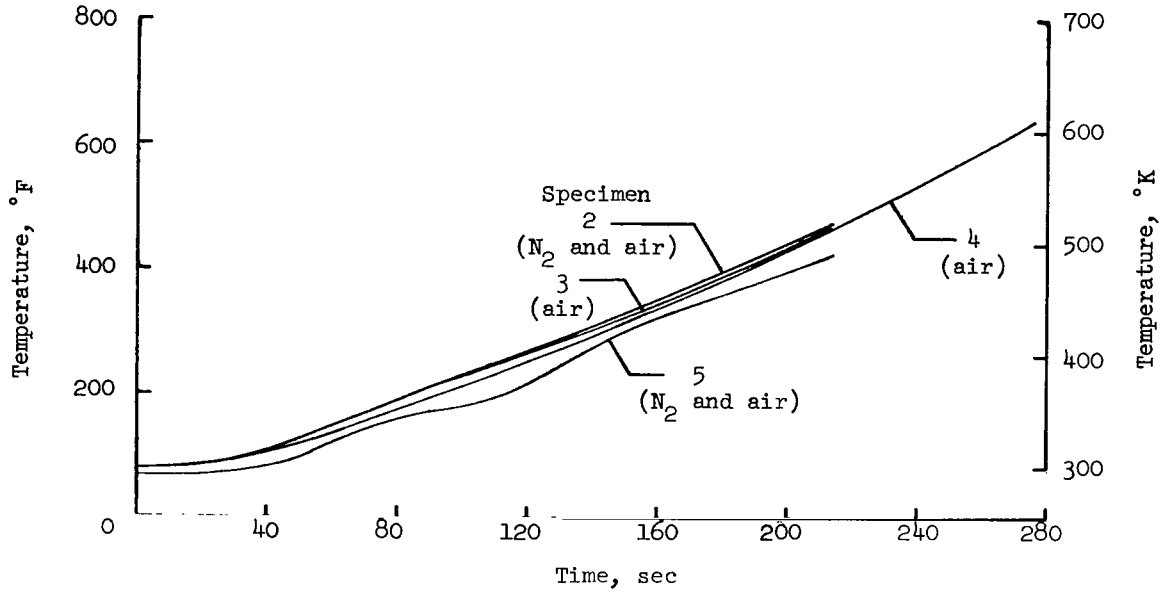
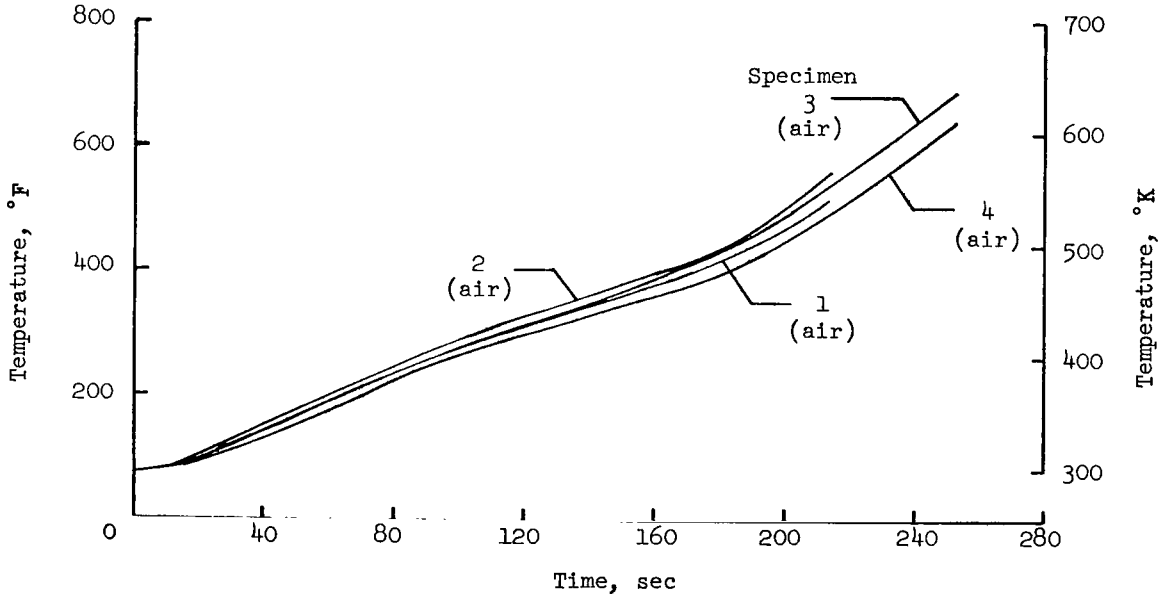


Figure 6.- Testing of arc-jet specimen. L-62-7174.1

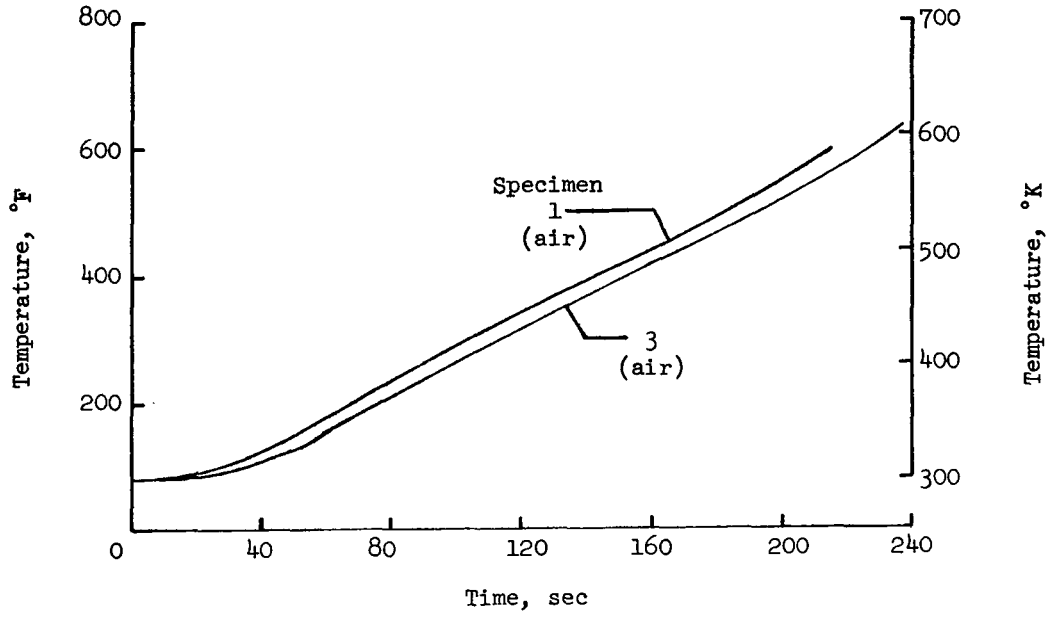


(a) Phenolic nylon.

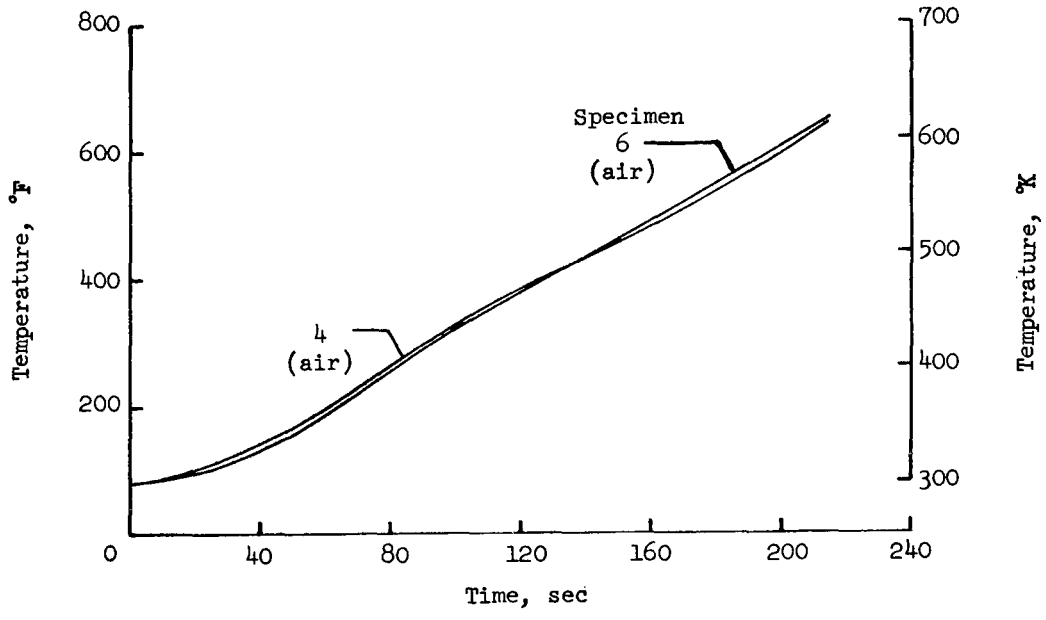


(b) Material A.

Figure 7.- Temperature histories of specimens tested in arc-jet facility.

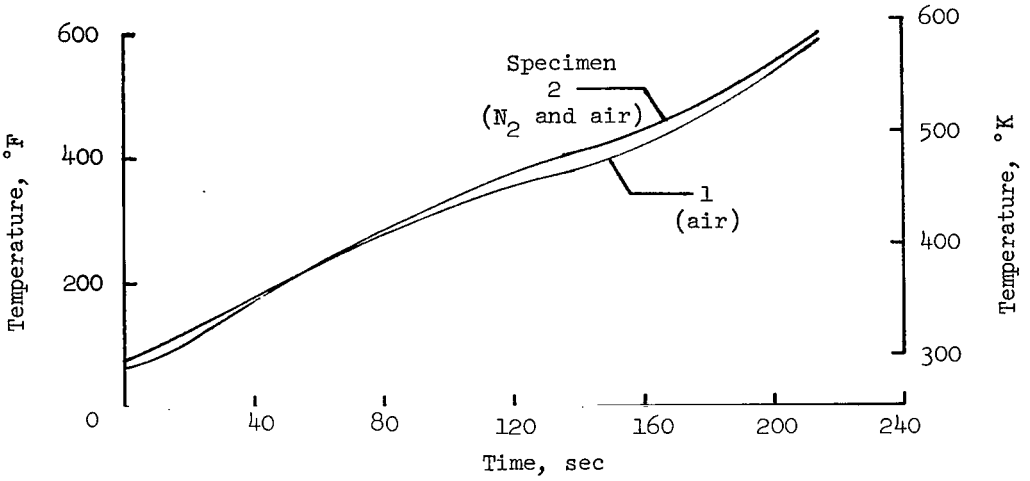


(c) Material B.

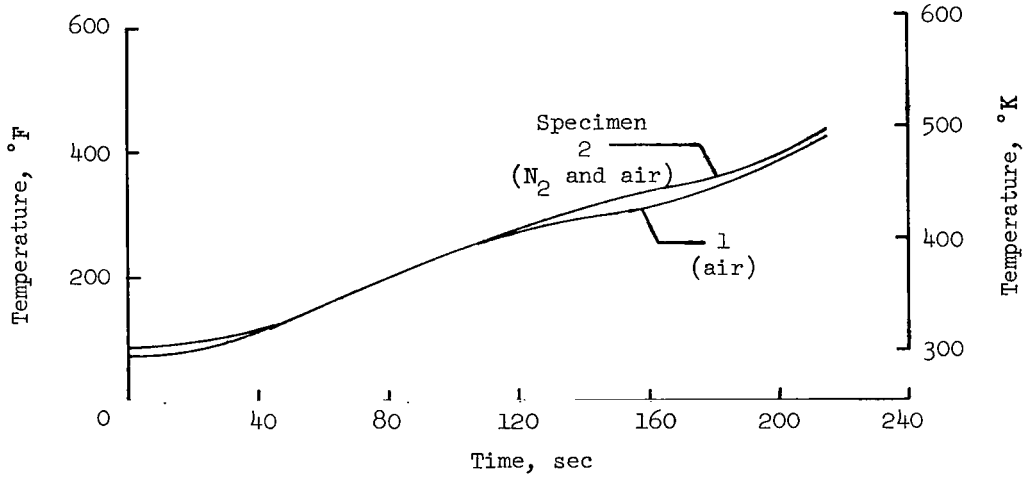


(d) Material C.

Figure 7.- Continued.

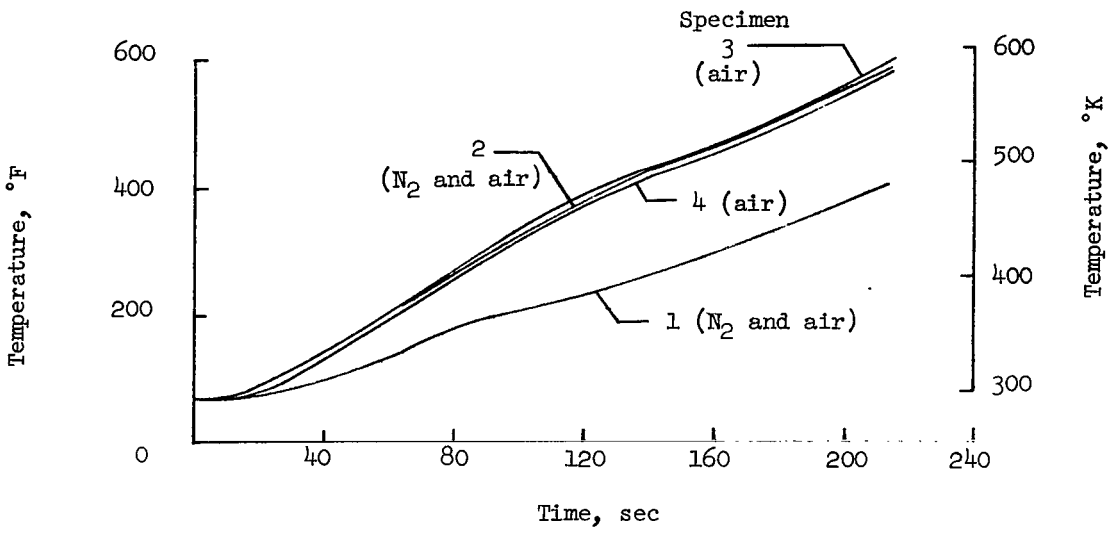


(e) Material D.

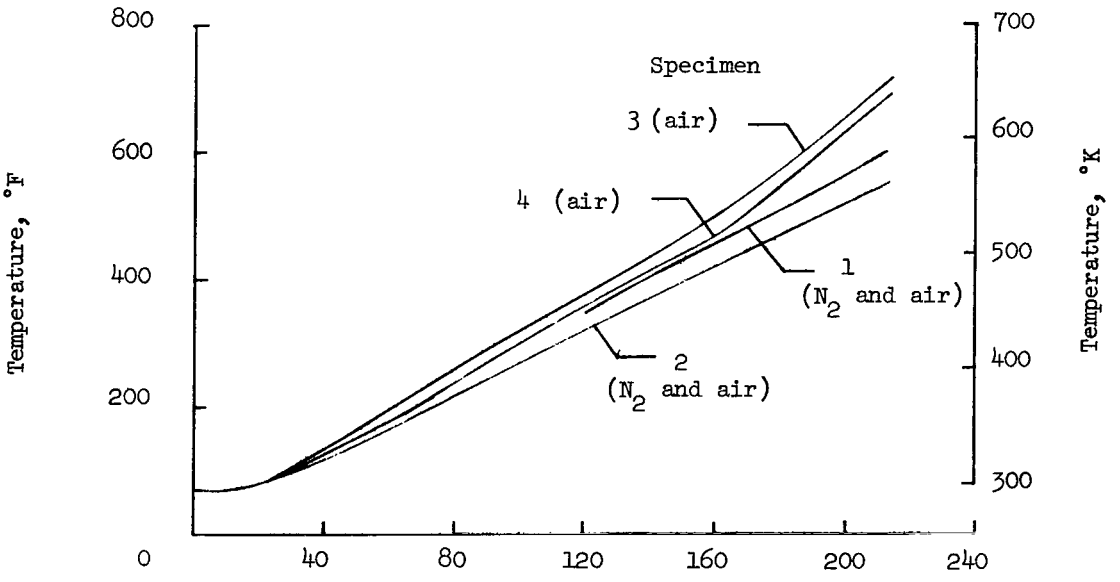


(f) Material E.

Figure 7.- Continued.

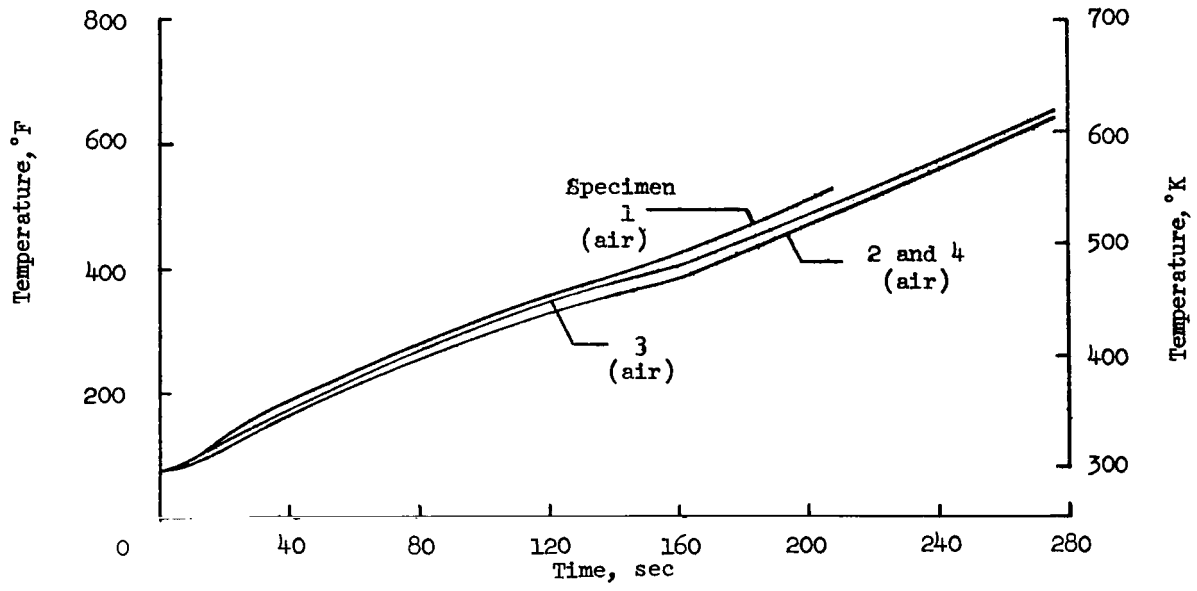


(g) Material F.

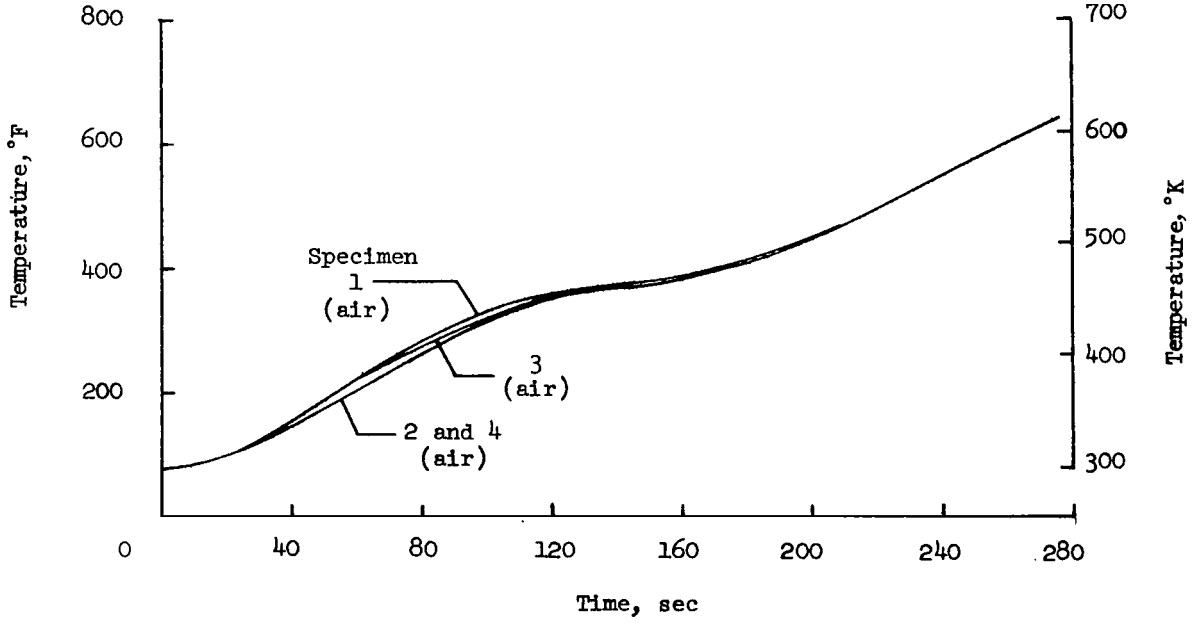


(h) Material G.

Figure 7.- Continued.

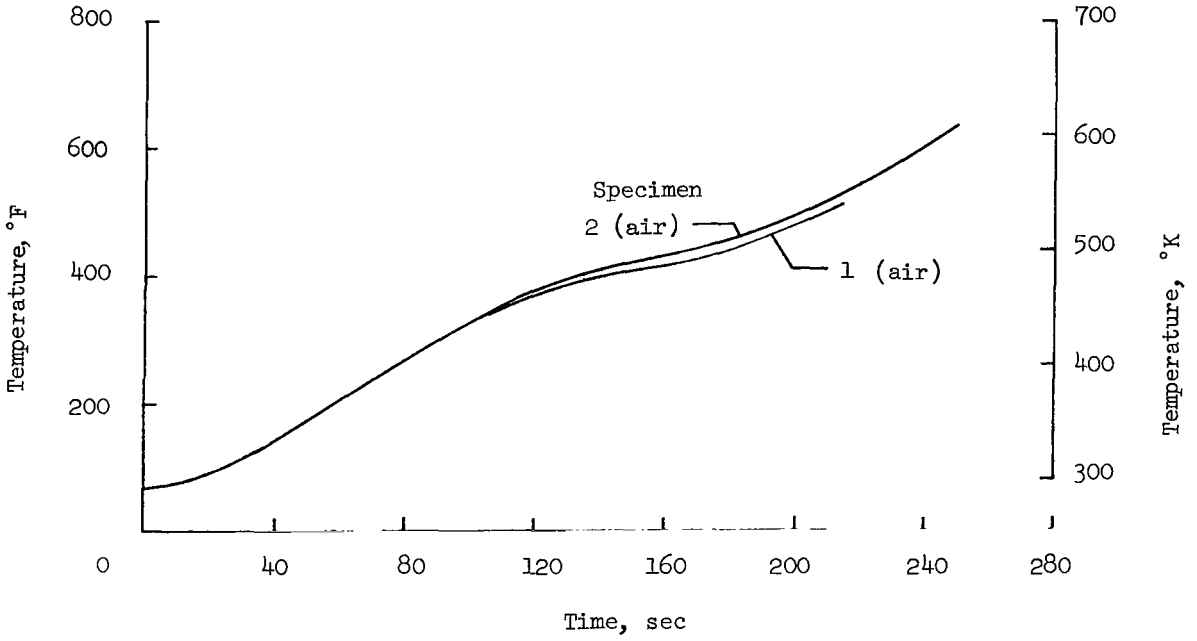


(i) Material H.



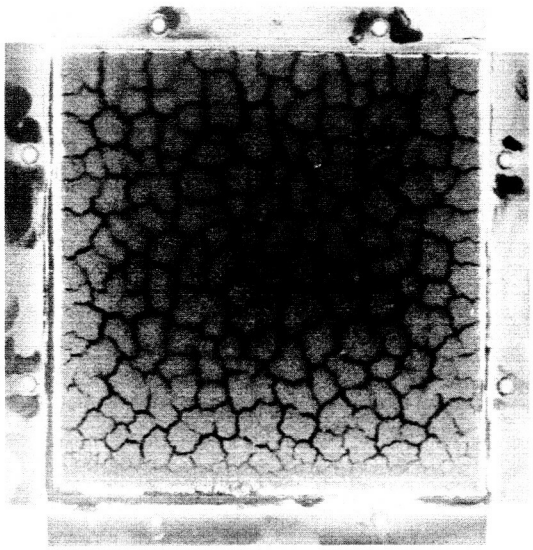
(j) Material I.

Figure 7.- Continued.



(k) Material J.

Figure 7.- Concluded.

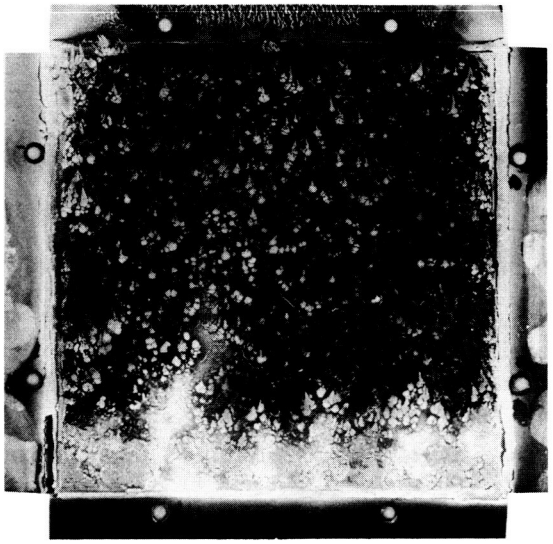


(a) Phenolic nylon.

Stream direction

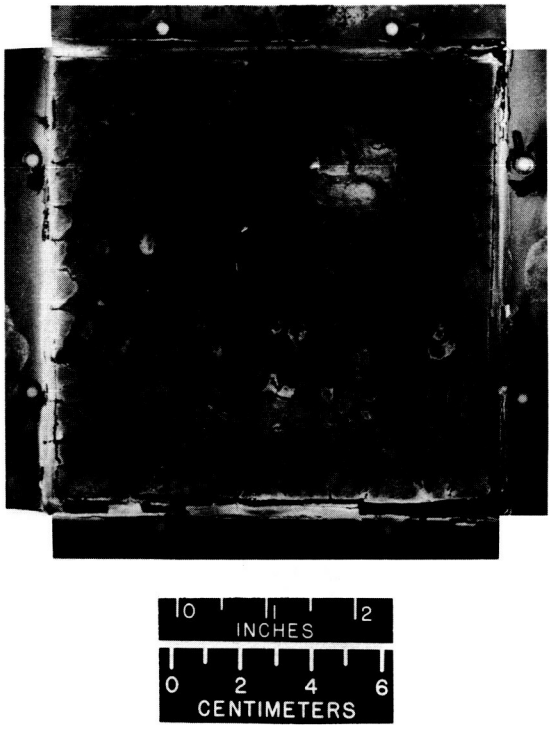
(b) Material A. L-64-8359

Figure 8.- Arc-jet specimens after testing.



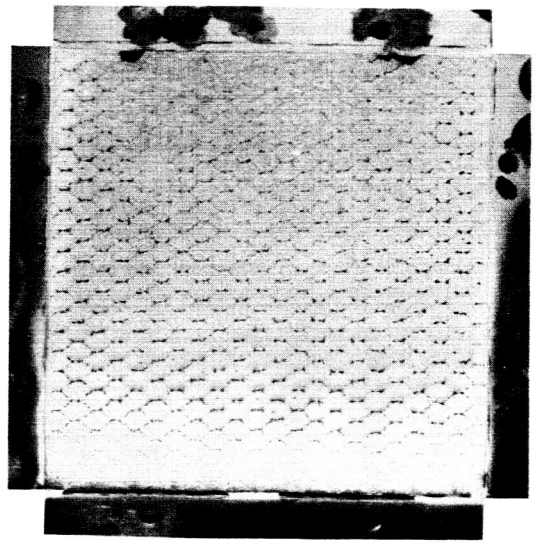
(c) Material B.

Stream direction



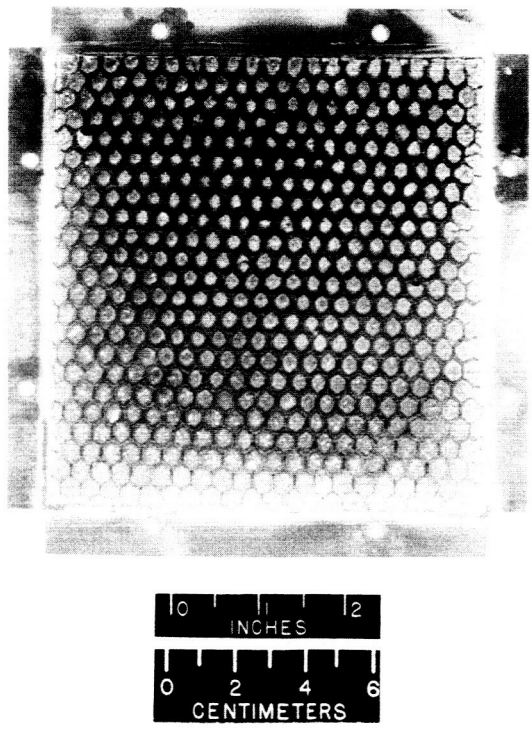
(d) Material C. L-64-8360

Figure 8.- Continued.



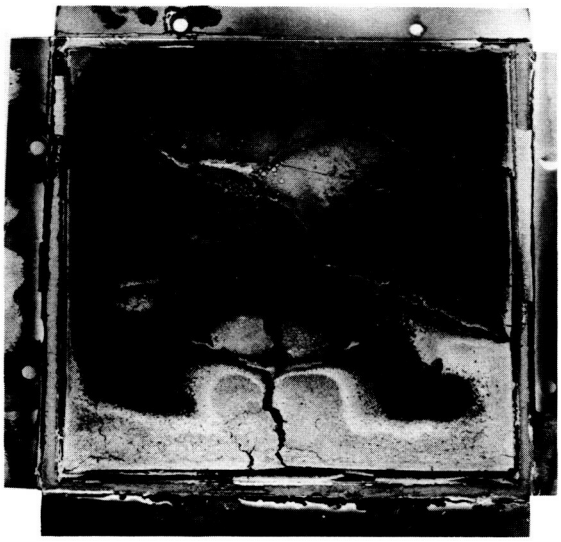
(e) Material D.

Stream direction



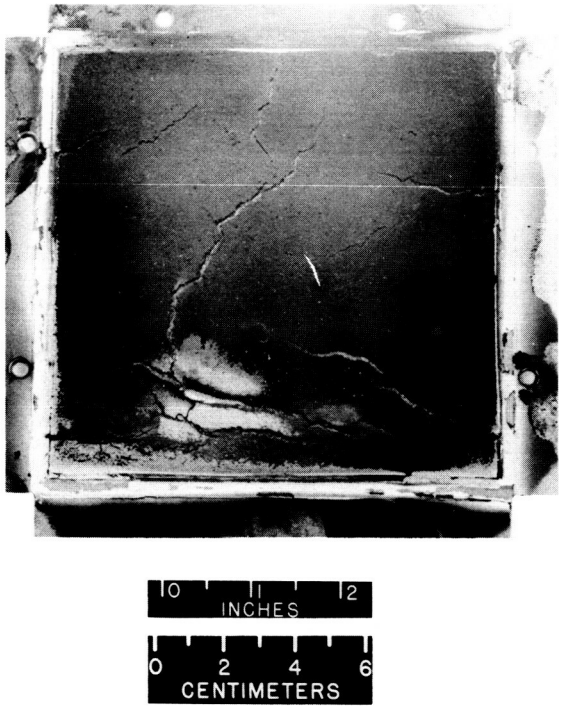
(f) Material E. L-64-8361

Figure 8.- Continued.



(g) Material F (tested in air).

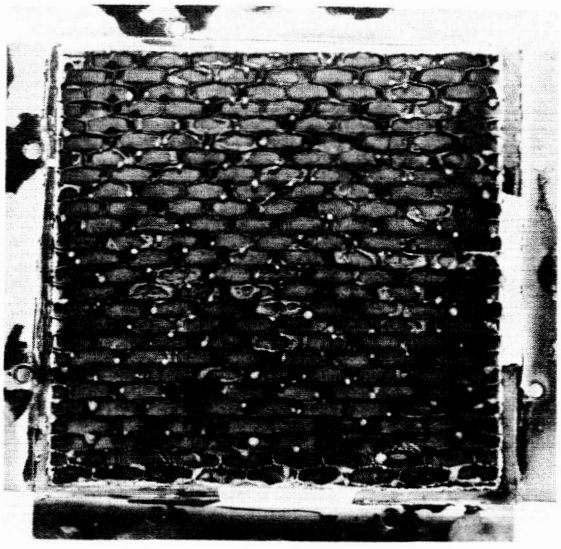
Stream direction



L-64-8362

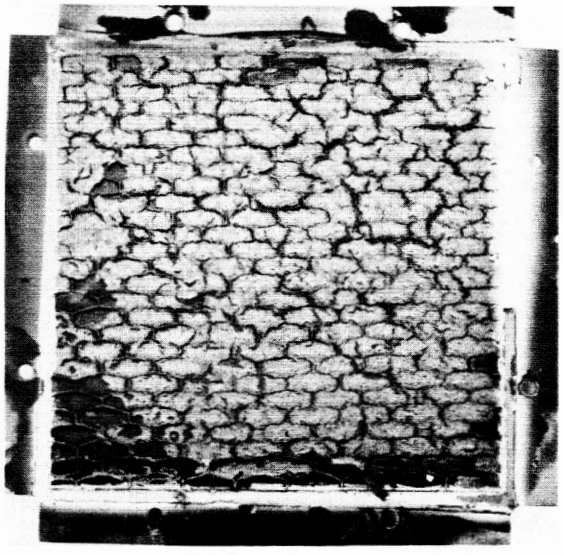
(h) Material F (tested in air and nitrogen).

Figure 8.- Continued.



(i) Material G (tested in air).

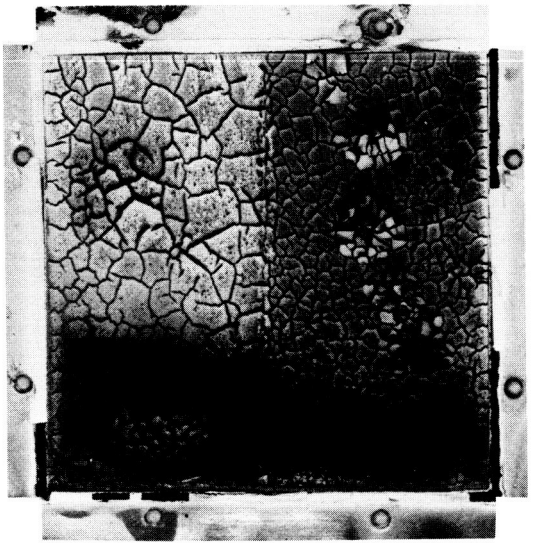
Stream direction



L-64-8363

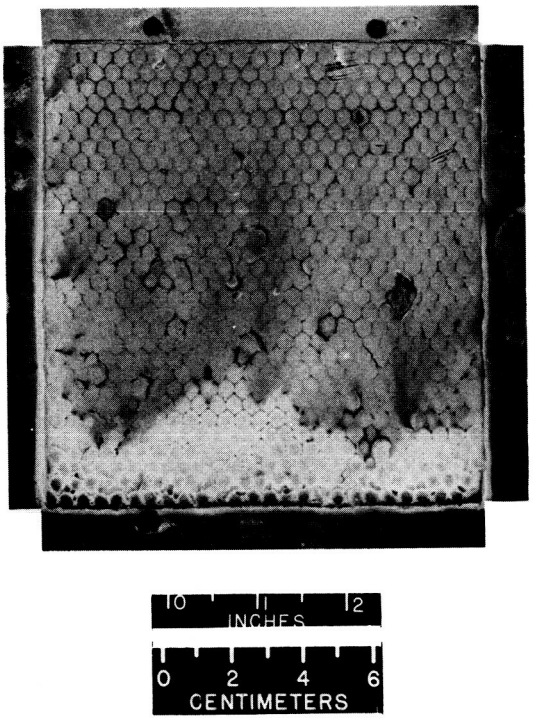
(j) Material G (tested in air and nitrogen).

Figure 8.- Continued.



(k) Material H.

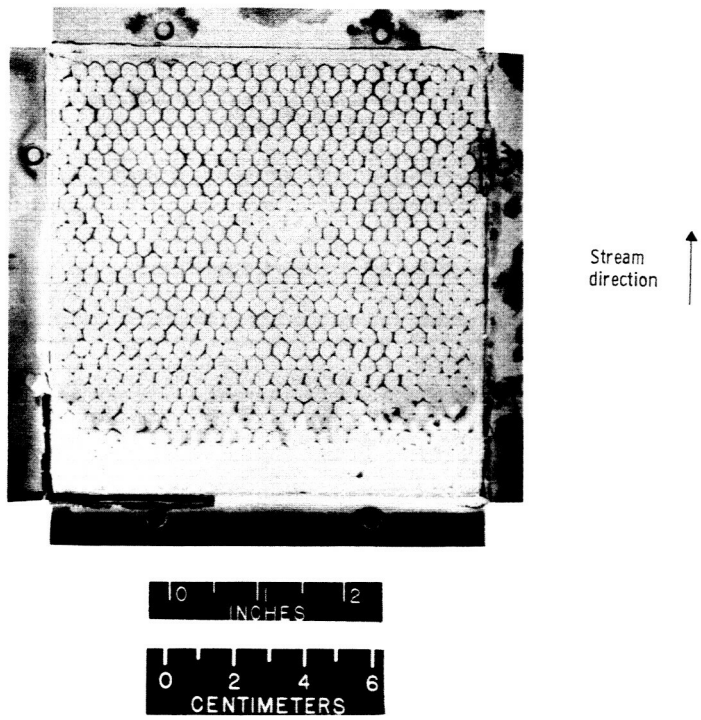
Stream direction



(l) Material I.

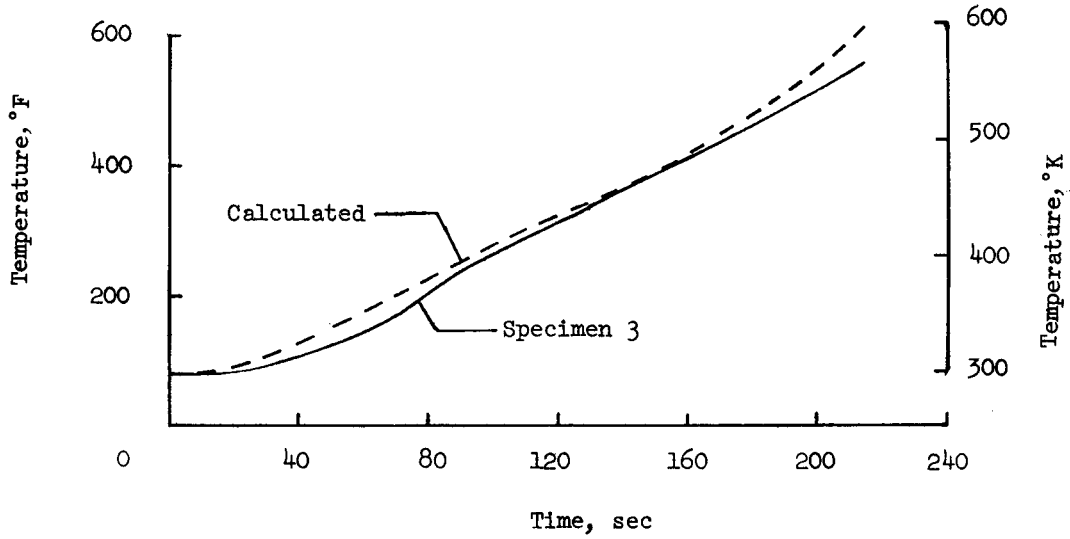
L-64-8364

Figure 8.- Continued.

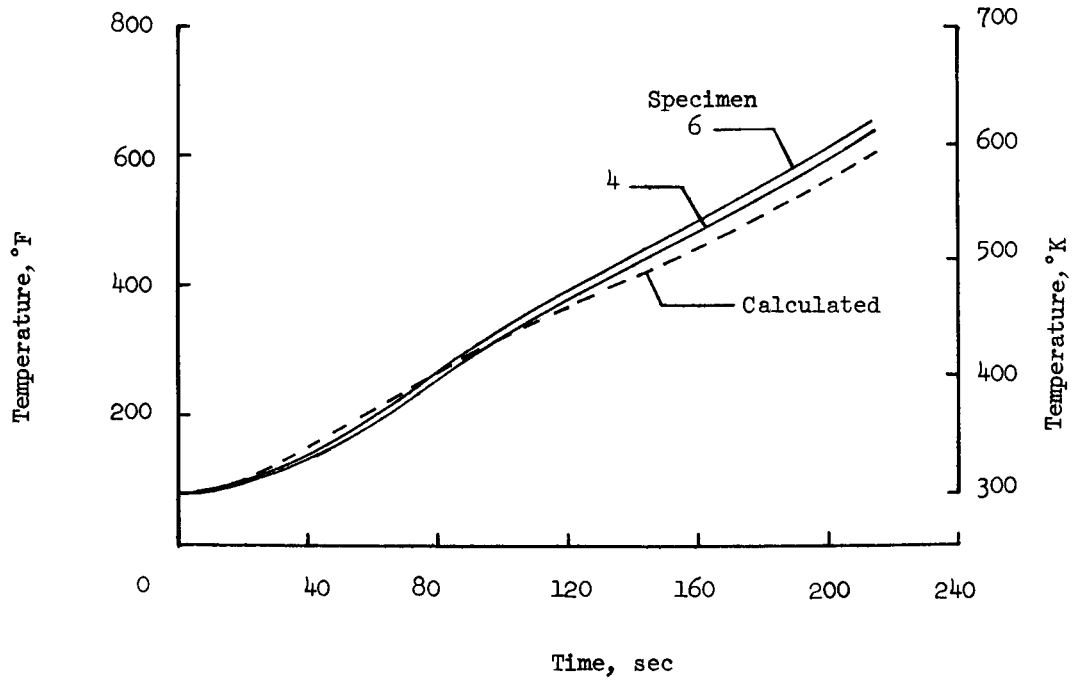


(m) Material J. L-63-736.1

Figure 8.- Concluded.

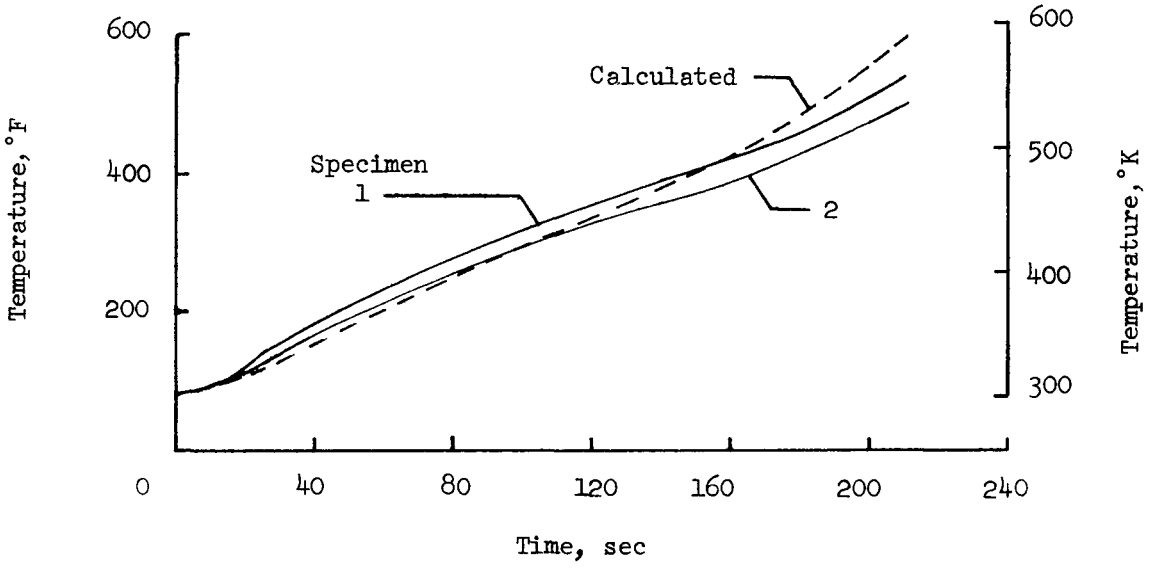


(a) Material B.

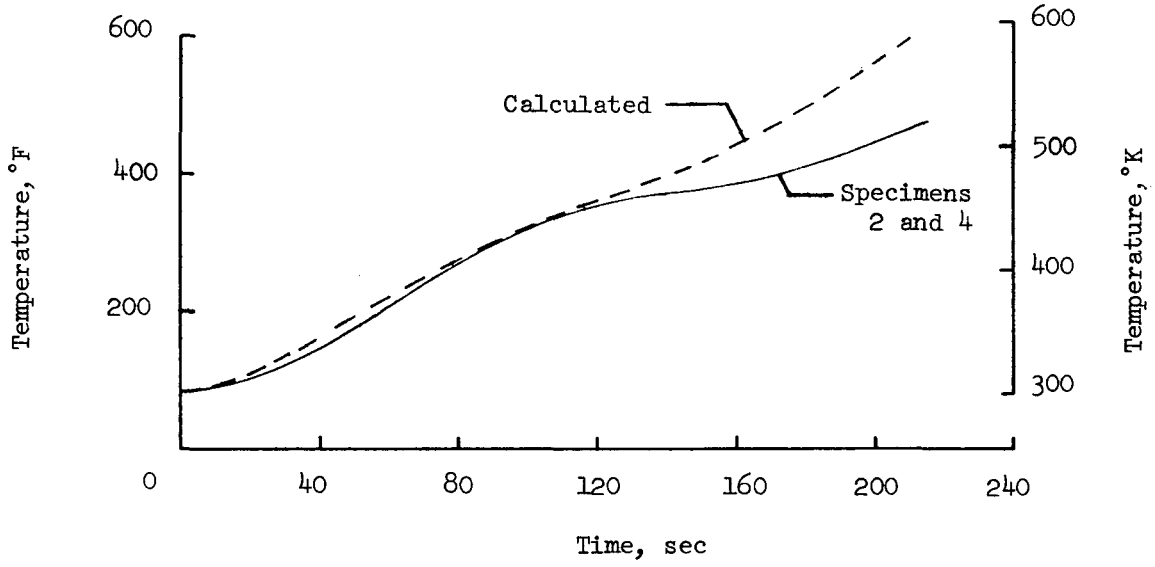


(b) Material C.

Figure 9.- Comparison of measured and calculated back-surface temperatures.

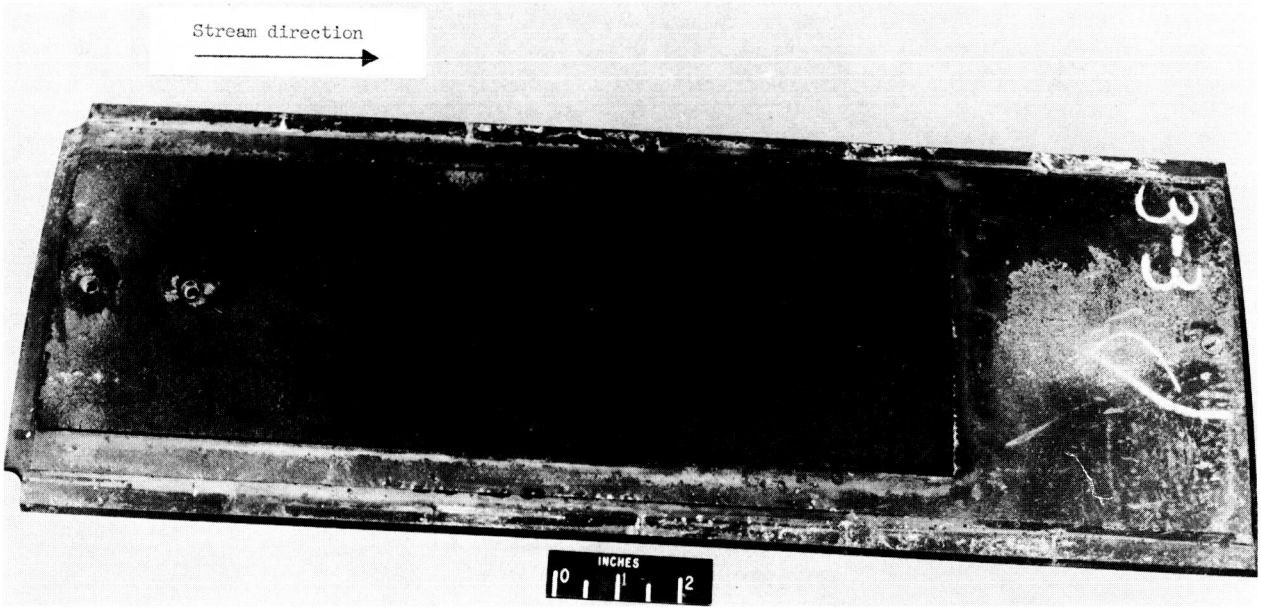


(c) Material H.

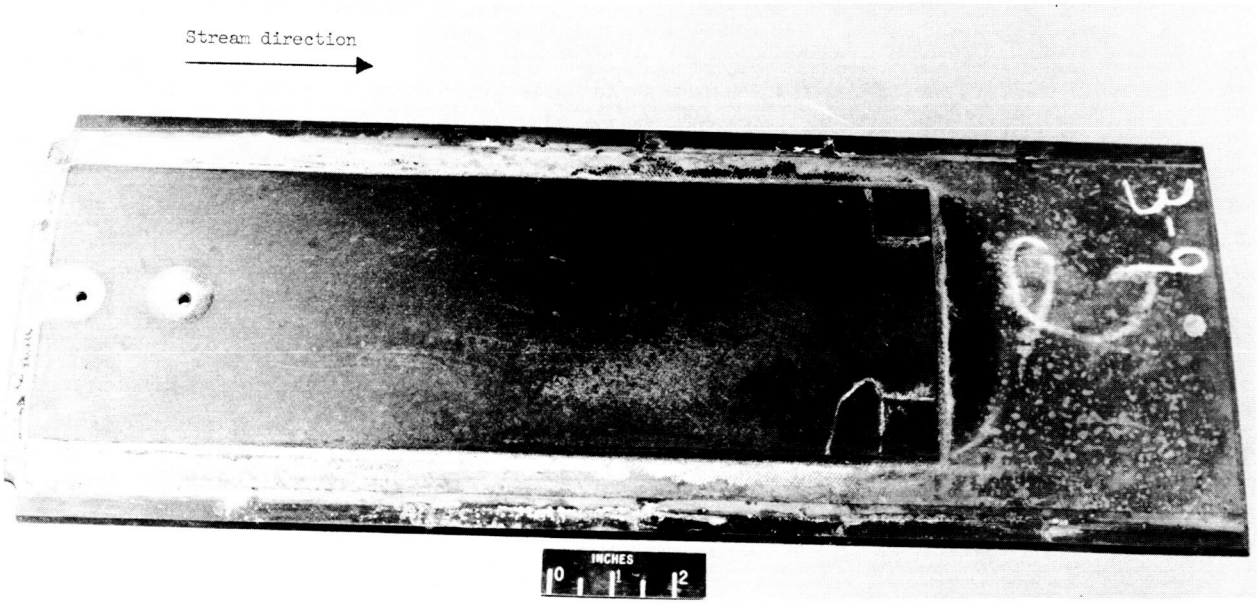


(d) Material I.

Figure 9.- Concluded.

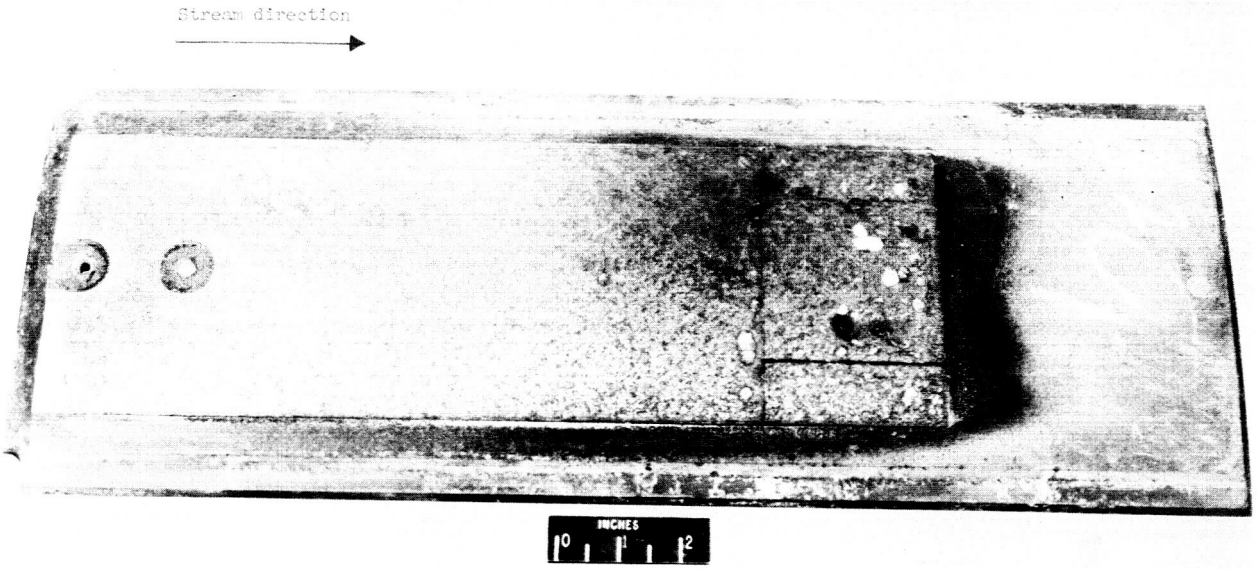


(a) Phenolic nylon. L-62-8458.1

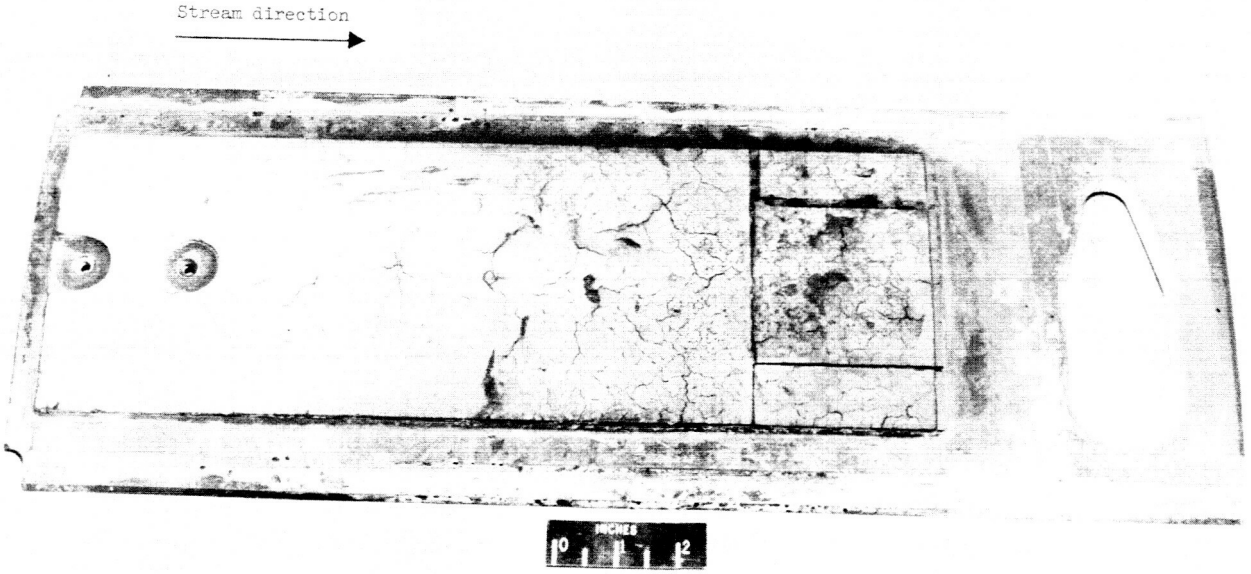


(b) Material A. L-62-8453.1

Figure 10.- Flight-test specimens after reentry.

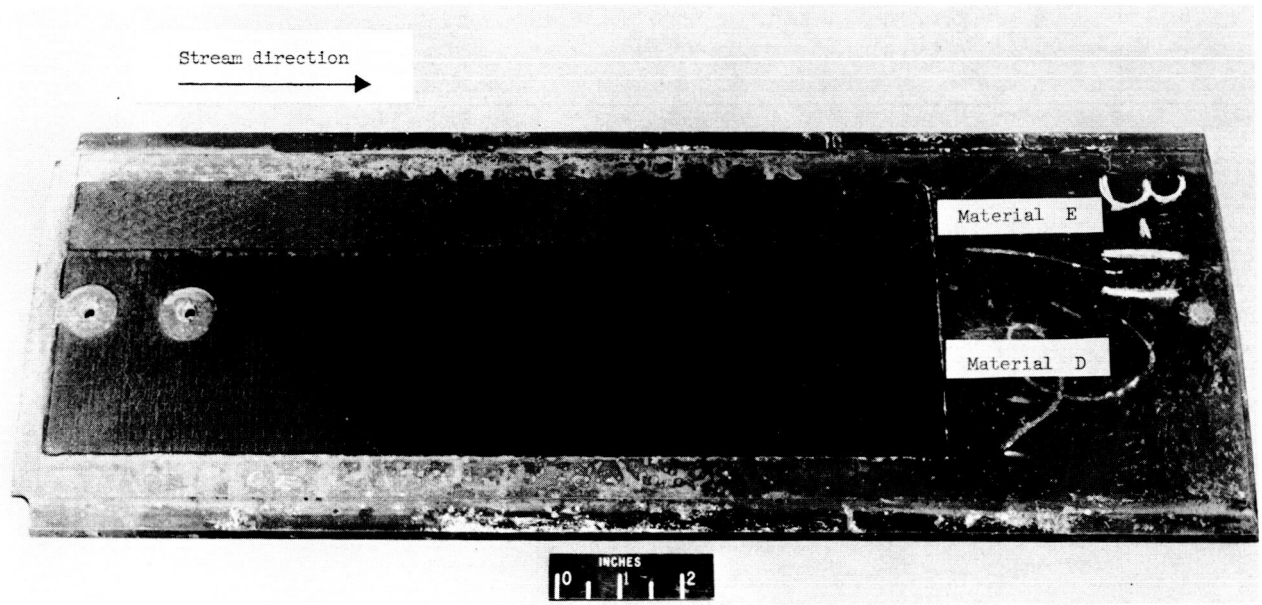


(c) Material B. L-62-8456.1

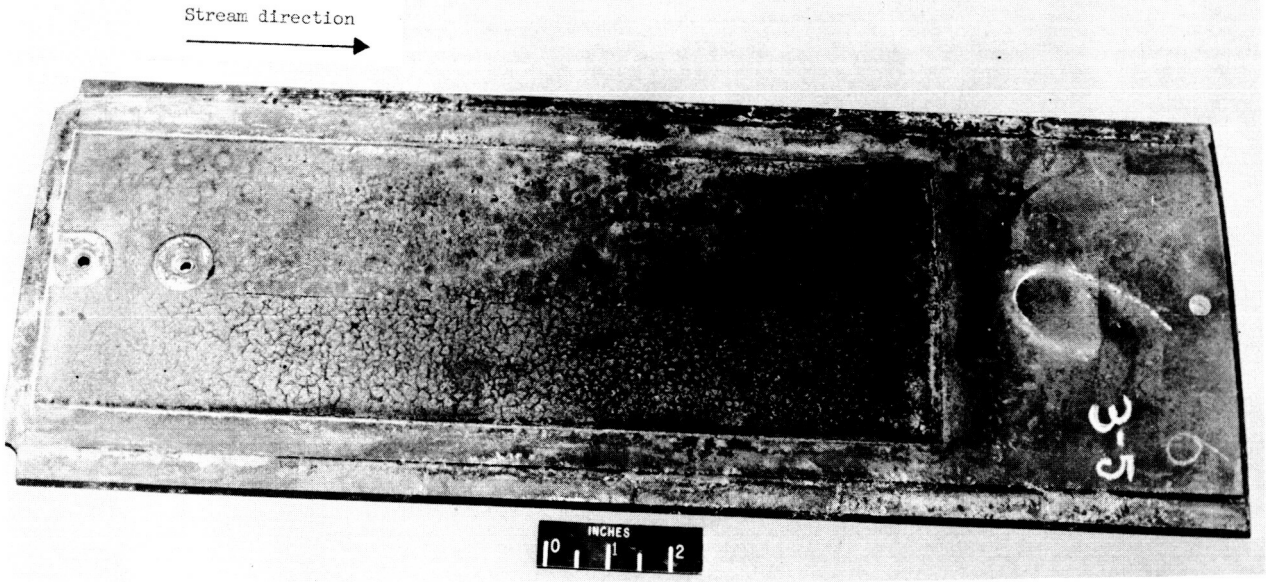


(d) Material C. L-62-8457.1

Figure 10.- Continued.

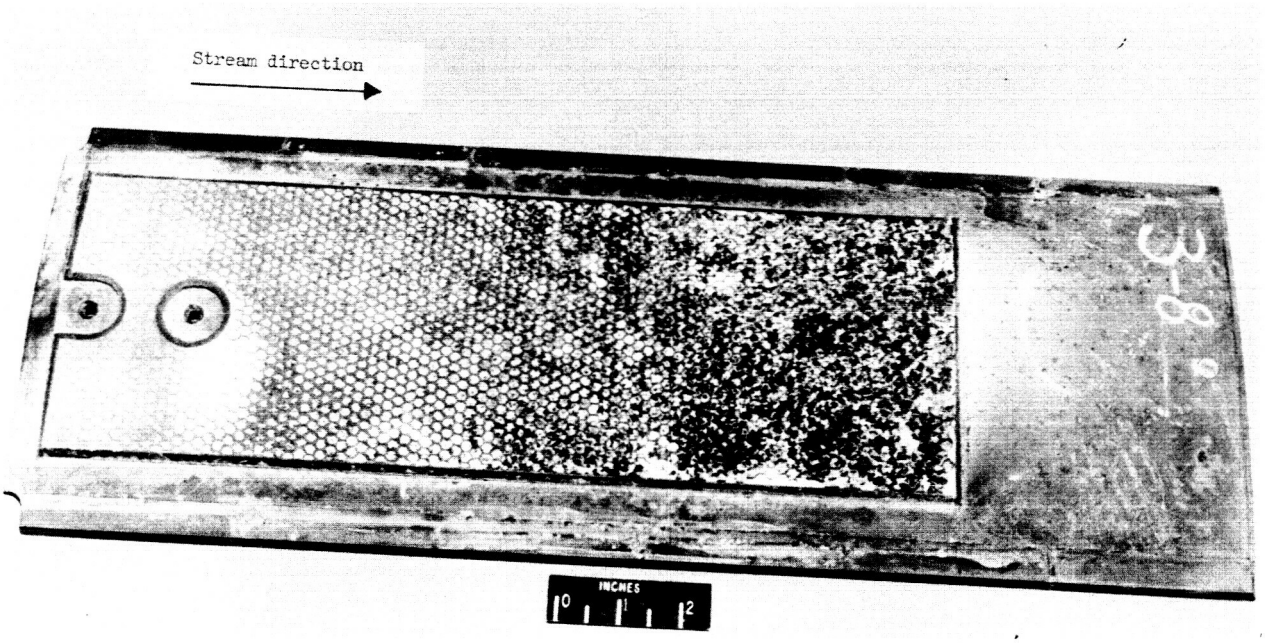


(e) Materials D and E. L-62-8452.1



(f) Material H. L-62-8460.1

Figure 10.- Continued.



(g) Material I. L-62-8455.1

Figure 10.- Concluded.

"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS: Information receiving limited distribution because of preliminary data, security classification, or other reasons.

CONTRACTOR REPORTS: Technical information generated in connection with a NASA contract or grant and released under NASA auspices.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

TECHNICAL REPRINTS: Information derived from NASA activities and initially published in the form of journal articles.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities but not necessarily reporting the results of individual NASA-programmed scientific efforts. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION DIVISION

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546